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RADIOLOGICAL WORK PLAN
FOR
THE NAVAL AIR STATION
JACKSONVILLE, FLORIDA

Prepared for

DEPARTMENT OF THE NAVY
SOUTHERN DIVISION
NAVAL FACILITIES ENGINEERING COMMAND

Under Contract No. N62467-93-D-0936

Prepared by

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FOREWORD

This Radiological Remediation Work Plan (RRWP) has been prepared to document the scoping and planning process performed by the U.S. Navy to support radiological remedial action activities at the Naval Air Station (NAS) Jacksonville, Florida Site. NAS Jacksonville is on the National Priorities List. A negotiated, signed Federal Facilities Agreement is in place for the site. Under the Comprehensive Environmental Response, Compensation, and Liability Act, a remedial investigation/feasibility study must be conducted to support the decision-making process for evaluating remedial action alternatives. The Site Management Plan for NAS Jacksonville includes a Remedial Investigation (RI) Report to be submitted for approval to the Environmental Protection Agency, Region IV, and the Florida Department of Environmental Protection.

Remedial action is necessary in selected areas of radiological contamination for the protection of human health and the environment. The remedial action will also support the completion of the Southern Division, Naval Facilities Engineering Command RI. This RRWP describes the approach that will be used to conduct the radiological remedial action and describes the organization that will be employed.

1.0 INTRODUCTION

The U.S. Department of Navy, Southern Division, Naval Facilities Engineering Command intends to conduct radiological remedial actions at the Naval Air Station (NAS) Jacksonville, Florida Site. Eighteen areas of the site (Figure 1-1) have been identified as radiological potential sources of contamination (PSCs). Bechtel Environmental, Inc. (BEI), the Environmental Response Action Contractor, will perform the radiological remedial action on these PSCs. This remedial action is required to support the Comprehensive Long-Term Environmental Action Contractor, ABB-Environmental Services, Inc. (ABB-ES) during completion of the remedial investigation.

This Radiological Remediation Work Plan (RRWP) is intended to document the scope of the radiological remediation effort and the procedures to be used.

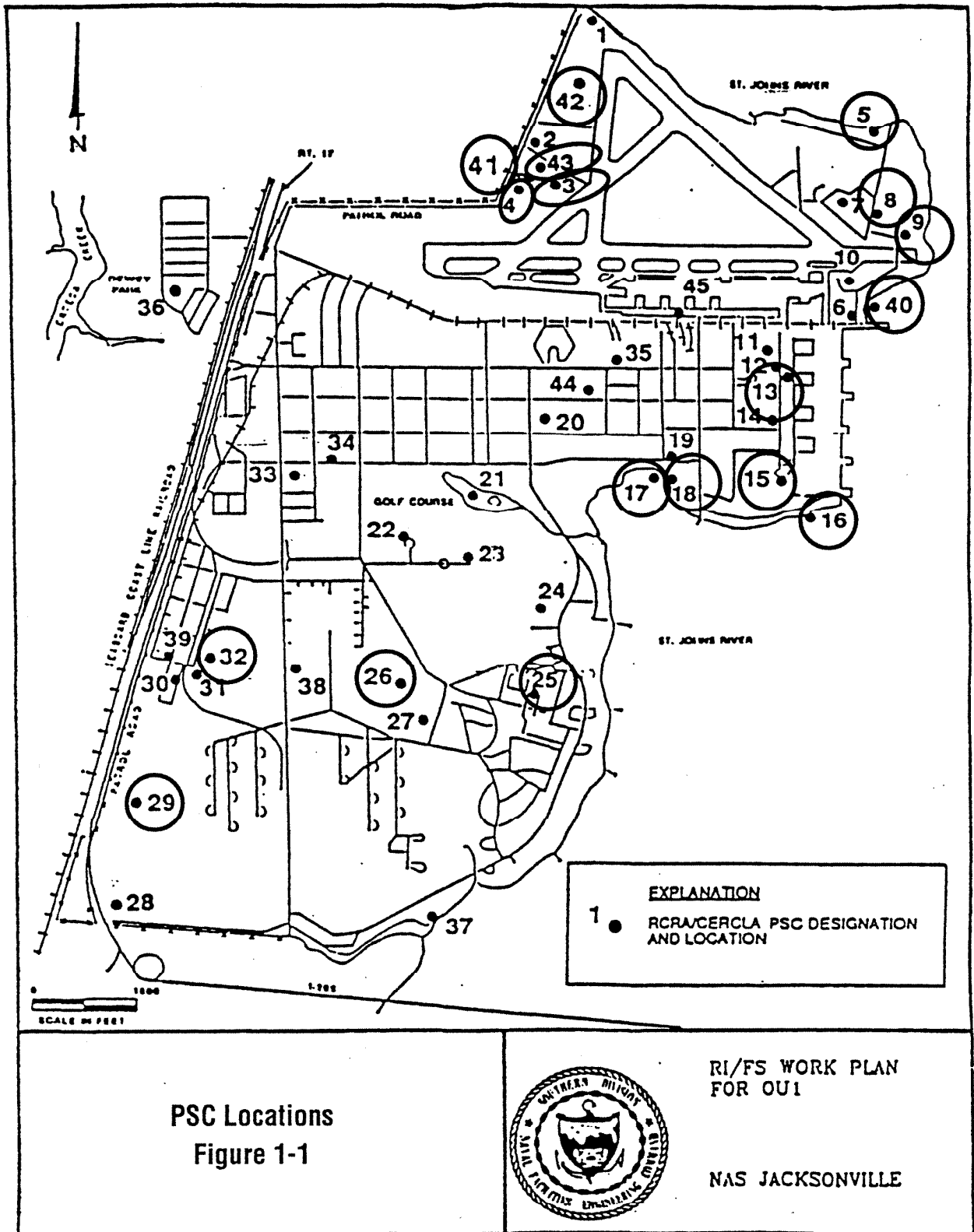
The activities described in this Plan are based on the following:

- the Department of the Navy Radiological Affairs Support Office (RASO) report of the technical assistance visit to the waste disposal sites at NAS Jacksonville, January 27 to February 2, 1993;
- observations made during a site visit by BEI, ABB-ES, and Navy personnel to NAS Jacksonville on March 31, 1994;
- discussion and conclusions from a meeting held between BEI, ABB-ES, and Navy personnel in Jacksonville on April 1, 1994;
- the specification for radiological survey at NAS Jacksonville, as prepared by ABB-ES, dated December 6, 1993;
- NAS Jacksonville's Radiological Survey and Remediation RAM Finalization Interface Meeting Minutes, Charleston, SC, July 8, 1994; and
- letter from Steve Watson, ABB-ES, to Dana Gaskins, Southern Division Naval Facilities Engineering Command, dated July 7, 1994.

In implementing this Plan, BEI will supply qualified personnel and equipment to the project; coordinate, manage, and supervise construction activities on site; assure compliance with contract and regulatory requirements; and lastly, provide documentation to the Navy that will include a data summary for ABB-ES's use in completing the RI report. BEI's approach to complete these tasks is presented in the following sections of this Plan.

The remainder of Section 1.0 provides general site information and the justification and objectives for the proposed radiological remediation.

Section 2.0 presents the BEI organization and responsibilities for completing the work. Section 3.0 provides a site history and a detailed description of the PSCs that have been identified by the Navy as requiring radiological surveys and remediation.



Section 4.0 provides the scope of work, the approach BEI will take to achieve the remedial objectives, and a description of the remedial action components and field activities.

Section 5.0 presents BEI's sampling and analysis plan and includes details on the radiological surveys and remediation activities planned.

Sections 6.0, 7.0, and 8.0 address the Waste Management Plan, Safety and Health Plan, and Quality Control Plan, respectively.

1.1 GENERAL SITE INFORMATION

NAS Jacksonville is located in southern Duval County approximately nine miles south of downtown Jacksonville. The site encompasses approximately 3,800 acres on the west bank of the St. Johns River, upstream from downtown Jacksonville. The primary mission of the site is to provide a work place, service, and managerial support for the operation and maintenance of naval weapons and aircraft to activities and units of the operating forces as designated by the Chief of Naval Operations (CNO).

The CNO and the Naval Energy and Environmental Support Activity conducted a preliminary assessment (PA) to evaluate the potential threat to human health on and off site from PSCs. The PA was performed as part of the Navy Assessment and Control of Installation Pollutants program, whose objective was to identify, assess, and control environmental contamination from past hazardous materials storage, transfer, processing, and disposal operations.

1.2 JUSTIFICATION AND OBJECTIVES FOR THE PROPOSED ACTION

The primary threat to human health and the environment associated with the radiologically identified PSCs is related to the potential for uncontrolled releases of contaminants from exposed surfaces and subsurface disposal areas. Contaminants could be released from these sources via infiltration and percolation, wind dispersion, gaseous emissions, runoff, leaching to groundwater, and disturbance by humans or animals. Direct exposure to gamma rays at the site is also a possibility. Even though the radioactive contamination at NAS Jacksonville does not pose an immediate threat to human health, the cleanup of uncontained materials is necessary for the long-term protection of human health and the environment.

2.0 ORGANIZATION AND RESPONSIBILITIES

2.1 PROJECT ORGANIZATION

BEI is the Environmental Response Action Contractor for the Navy, Southern Division Naval Facilities Engineering Command. BEI is teamed with General Engineering Laboratories (GEL), who will perform the radiological analyses required for NAS Jacksonville. A project organization chart is provided in Figure 2-1.

2.2 COORDINATION AND RESPONSIBILITIES FOR FIELD WORK

As the Environmental Response Action Contractor for the Navy, BEI provides support to the NAS Jacksonville RI and management of remedial action field activities, which includes all

activities necessary to implement field work delineated in work plans. Typically, these activities include development and procurement of subcontract services; development, implementation, and overview of plans; collection and review of data, including sampling results, quality assurance/quality control submittals, and sample tracking and custody; technical guidance to onsite personnel; report preparation; cost management; and schedule control.

The BEI program manager is responsible to the Navy for the completion of all aspects of the work. The program manager is supported by a project manager and representatives from engineering, construction, environmental safety and health, contract administration, quality control, project administration, and project controls. A brief description of the responsibilities of the project manager and each group are described below.

2.2.1 Project Manager

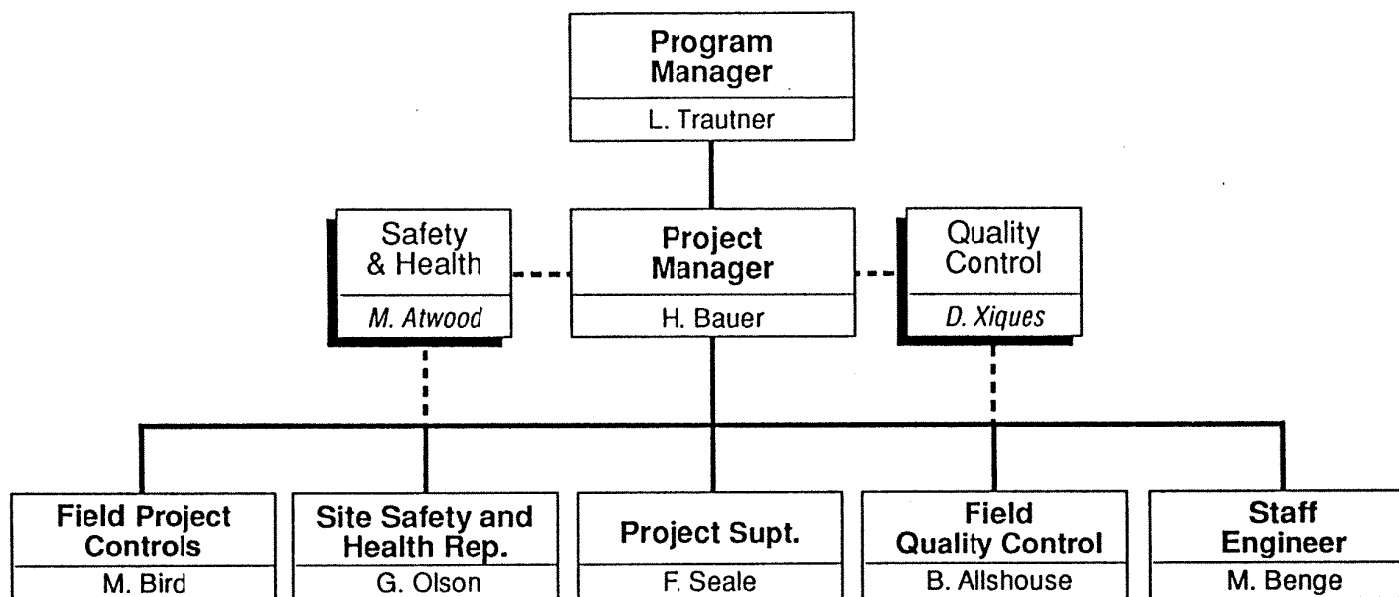
- Implements overall guidance provided by the BEI program manager on a site specific basis
- Manages a team of professionals from each of the disciplines described below to accomplish the goals of the Naval Facilities Engineering Command project managers and the BEI program manager
- Interfaces directly with Navy project managers to implement directions on a site-specific basis

2.2.2 Engineering

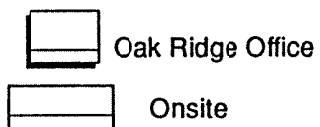
- Develops bid packages and technical specifications needed to subcontract any remedial action work
- Provides site interface/coordination with regulatory agencies
- Modifies technical specifications and drawings, as required
- Provides geotechnical field support to remedial action efforts
- Provides onsite waste management and identification
- Participates in technology selection
- Develops work plans for remedial action
- Manages and evaluates chemical and radiological data obtained during remedial action activities

2.2.3 Construction

- Reviews all site plans for constructibility
- Provides field engineering services to monitor onsite work



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Figure 2-1
Project Organization

- Administers subcontracts to complete work plans (i.e., cost, completion)
- Obtains manual craft
- Directs craft to implement work plans
- The project superintendent is responsible to the BEI project manager for day-to-day operations at the site.

2.2.4 Environmental Safety and Health

- Develops plans, objectives, evaluations, and documentation for all environmental compliance, safety, and health matters
- Ensures all applicable federal, state, and local regulatory requirements are met
- Supports onsite waste management
- Provides site-specific safety and health training
- Provides a site safety and health representative (SSHR)
- Performs audits of site activities to ensure implementation of the Safety and Health Plan and to assess the effectiveness of the program.

2.2.5 Contract Administration

- Identifies bidders for subcontract work
- Coordinates bid and subcontract bid and award process
- Manages revisions to subcontracts
- Ensures compliance with Prime Contract

2.2.6 Quality Control

- Prepares site-specific quality control (QC) plan
- Implements the QC plan
- Audits quality assurance system and performance
- Conducts periodic reviews of program plans

2.2.7 Project Controls

- Provides cost and schedule support, including budgeting and monitoring

- Provides site automation services

2.2.8 Project Administration

- Provides administrative services such as document control, reproduction, archival, and mail distribution
- Provides document editing services

3.0 SITE BACKGROUND AND SETTING

3.1 GENERAL SITE DESCRIPTION

The operating portion of NAS Jacksonville lies between U.S. Highway 17 and the St. Johns River; however, the site also occupies 515 acres (included in the site's 3,800 total acreage) west of U.S Highway 17. This RRWP focuses on the radiological PSCs, which are identified as PSCs 3, 4, 5, 8, 9, 13, 15, 16, 17, 18, 25, 26, 29, 32, 40, 41, 42, and 43. The total acreage of these 18 radiological PSCs is approximately 192 acres of land surface. In addition, PSCs 16, 17, and 40 are outfall locations and therefore are under water. PSC 42 is a former waste water treatment plant polishing pond and covers approximately 4.5 acres.

Of these PSCs, PSC 26 is the only one with restricted access. One building is planned for a radiological survey during this remediation effort. The PSCs of concern are open grounds where minimal clearing, to heavy clearing in some instances, will be required.

3.2 SITE HISTORY

The NAS Jacksonville site was originally designed for seaplane support, and when established in 1940, included facilities for pilot training and a Naval Aviation Trades School. At the end of World War II, NAS Jacksonville was devoted entirely to aviation training. The Naval Aviation Depot (NADEP) has been a major tenant of NAS Jacksonville since it operated as an assembly and repair department in 1940. The NADEP provides maintenance, engineering, logistic, and support services and performs rework, repair, and modification of aircraft, engines, and aeronautical components. Today, the NADEP is the largest tenant command on NAS Jacksonville and the largest industrial employer in Northeast Florida and Southeast Georgia, with a civilian work force of over 3,300 employees.

From June 28 to July 2, 1982, the onsite phase of a preliminary assessment (PA) was conducted as the first phase of the Naval Assessment and Control of Installation Pollutants program. This program has now been incorporated into the Navy and Marine Corps Installation Restoration (IR) program. The purpose of the PA was to identify, assess, and control environmental contamination from past hazardous materials storage, transfer, processing, and disposal operations. The PA identified 38 PSCs, of which 10 posed a potential threat to human health and the environment based on their Confirmation Study Ranking System (CSRS) scores. Two PSCs (PSCs 26 and 27) required interim remedial actions before conclusion of the PA.

CSRS scores and engineering judgement were used to determine the need for a Site Inspection (SI). The 10 PSCs judged to pose a potential threat to human health and the environment were

selected for the SI. The SI was conducted to determine the presence or absence of contamination in the soil, surface water, or ground water at each of the 10 PSCs. The SI was conducted in August 1983. In October 1983, the Florida Department of Environmental Regulation (FDER), now the Florida Department of Environmental Protection (FDEP), reviewed the PA and suggested four additional PSCs for investigation and additional investigation on PSC 29. Newly acquired information resulted in the addition of six more PSCs to the scope of the SI.

ABB-ES is presently conducting the RI for the Navy. BEI is supporting this RI with radiological remedial action.

3.3 RADIOLOGICAL CONDITIONS

Several PSCs have been identified as having been potentially exposed to sources of low-level radioactive material or radioactive contamination over the past fifty years. Sources of low level ionizing radiation in the form of luminous instrument dials are known to have been used at the site. The source of energy in the luminous dials was radium-226. Several of the shops at NAS Jacksonville were used to maintain, repair, store, or calibrate these gauges and dials.

Aircraft repair and maintenance operations in the past generated low level radioactive radium paint wastes, which were disposed onsite. Vacuum tubes and other aircraft instrument parts with ionizing radiation sources and luminous instrument dials were also disposed on site. Aircraft instrument dial stripping operations took place during and after World War II.

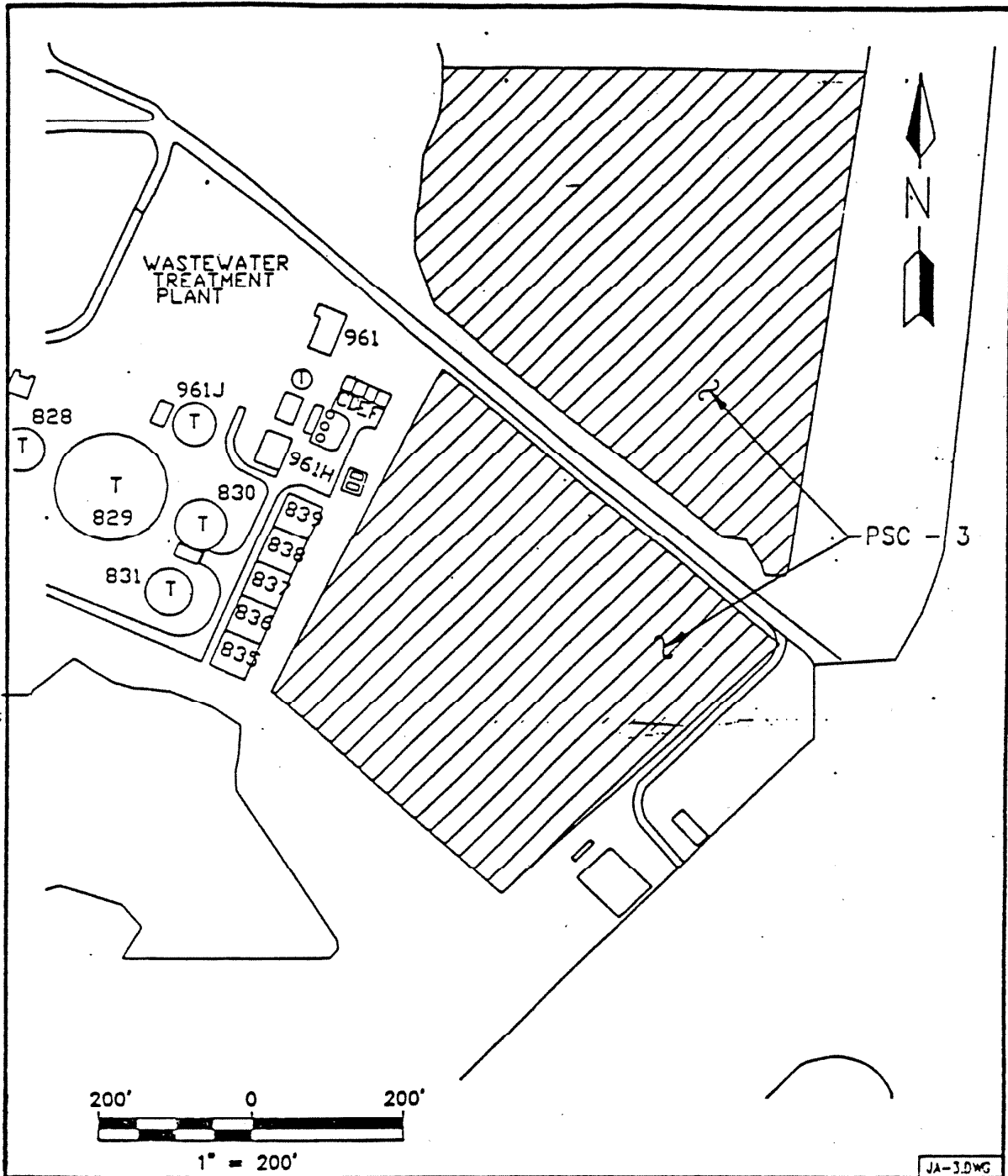
A removal action for buried radium-painted aircraft instrument dials was conducted on PSC No. 26. The Navy completed the site identification, removal action, and verification survey. Over 500 55-gallon drums of contaminated soil and point sources were cleaned up as a result of this effort. PSC 26 was released from radiological controls and returned to service after the verification survey.

In January 1993, through spot surveys, RASO discovered that additional sources of radioactive material may still be buried at PSC No. 26. Additional areas throughout NAS Jacksonville were also identified by RASO as needing a formal radiological survey to evaluate whether additional sources of radioactive contamination exist.

A brief description of each PSC identified by the Navy as requiring radiological investigation/remediation is provided below.

PSC No. 3: This PSC is the waste water treatment plant ex-sludge disposal area. It is located east of the sewage treatment plant, and comprises approximately 15 acres (Figure 3-1). Approximately 20,000 tons of sewage sludge containing metals and organics were dumped here from 1962 to 1980.

Descriptions of historical site operation coupled with previous radiological measurements indicate that the water treatment system may have been contaminated by radium paint disposal into the drain system from aircraft instrument maintenance and repair. Therefore, the PSCs that include waste water treatment systems will be treated as a single source for the purpose of radiological source characterization. The "West" wastewater treatment system includes PSC 3, 4,, 41, 42, and 43. This grouping will be treated separate from those PSCs associated with the "East"



PSC - 3
 WWTP EX-SLUDGE DISPOSAL AREA
 FIGURE 3-1



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wastewater treatment systems due to the time periods associated with operation of each. The characterization protocols will be based on definitions in Section 5 for a nonsuspect area.

PSC No. 4: PSC No. 4 is the pine tree planting area. It is located approximately 200 feet south of the sewage treatment plant (Figure 3-2) and extends approximately 3,600 feet west. This area was reportedly used for disposal of paint shavings, sewage sludge, asbestos, oil and other petroleum products. BEI will conduct radiological surveys at PSC No. 4, in combination with characterizing PSC 3, 41, 42, and 43.

PSC No. 5: This PSC is the shoreline fill located west of the fuel barge dock area. It was used in the 1940s for waste disposal that included dredge spoils from PSC-40. It is approximately 300 feet wide by 2,200 feet long (Figure 3-3). BEI will conduct radiological surveys at PSC No. 5, in combination with PSC-8, 16, and 40, according to protocols for nonsuspect areas as defined in Section 5. In the event that radiological contamination is detected and extends to the shoreline/water interface, sediment sampling offshore, in the St. Johns River will be conducted.

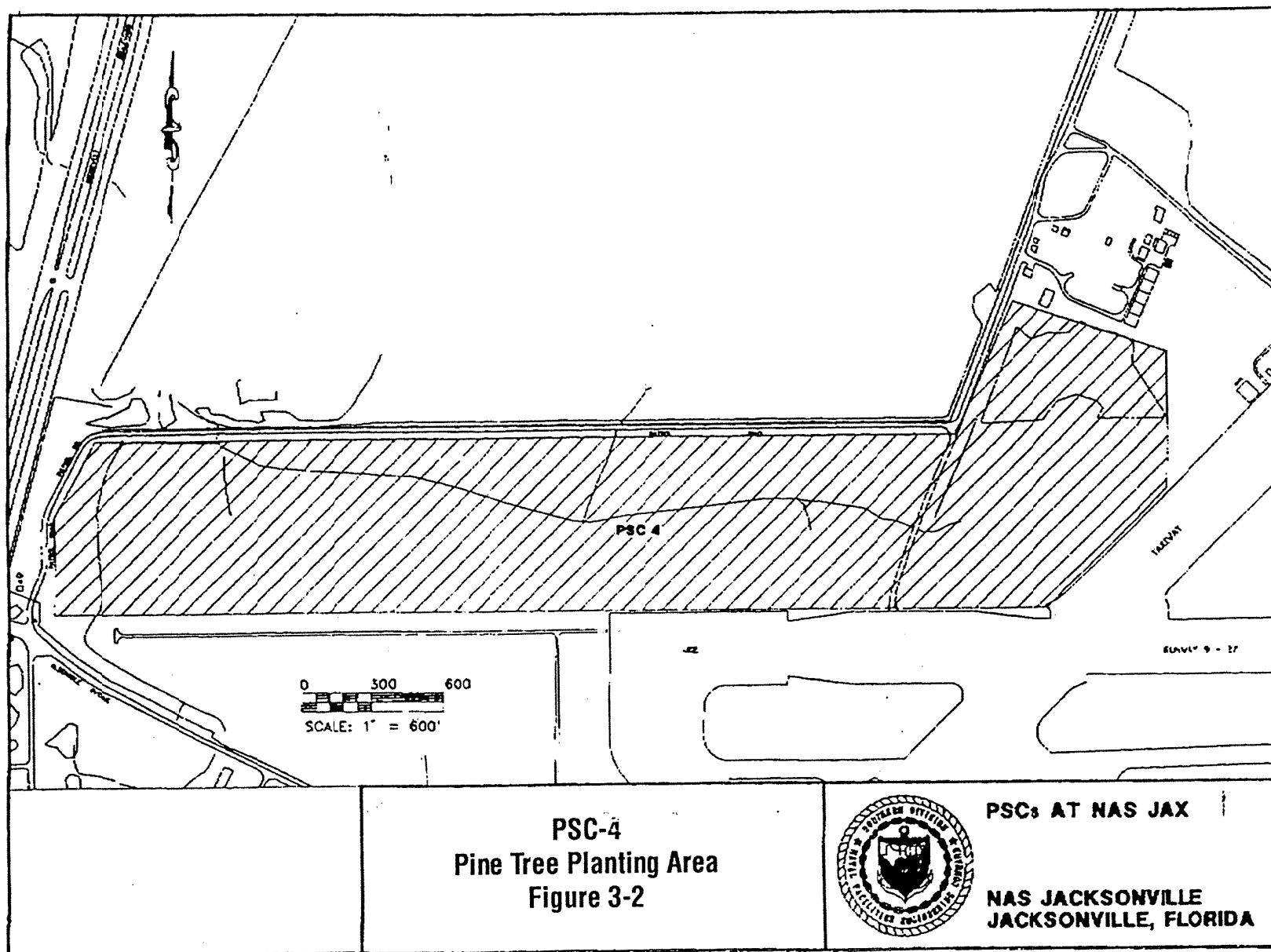
PSC No. 8: PSC No. 8 is a vacant lot, east of the fuel farm area, on the east side of Catapult Road (Figure 3-4). It covers approximately 4.7 acres. PSC No. 8 was used by the Navy for parking fighter planes. In addition, an inspection of the PSC revealed material, possibly abrasive blast grit, stored at this location. The Navy indicated that sediments dredged from the polishing and oxidation ponds were spread on PSC 8, as well as dredge spoils from PSC-40. BEI will conduct radiological surveys at PSC No. 8 in combination with PSC-5, 16, and 40, according to protocols for a nonsuspect area, as defined in Section 5.

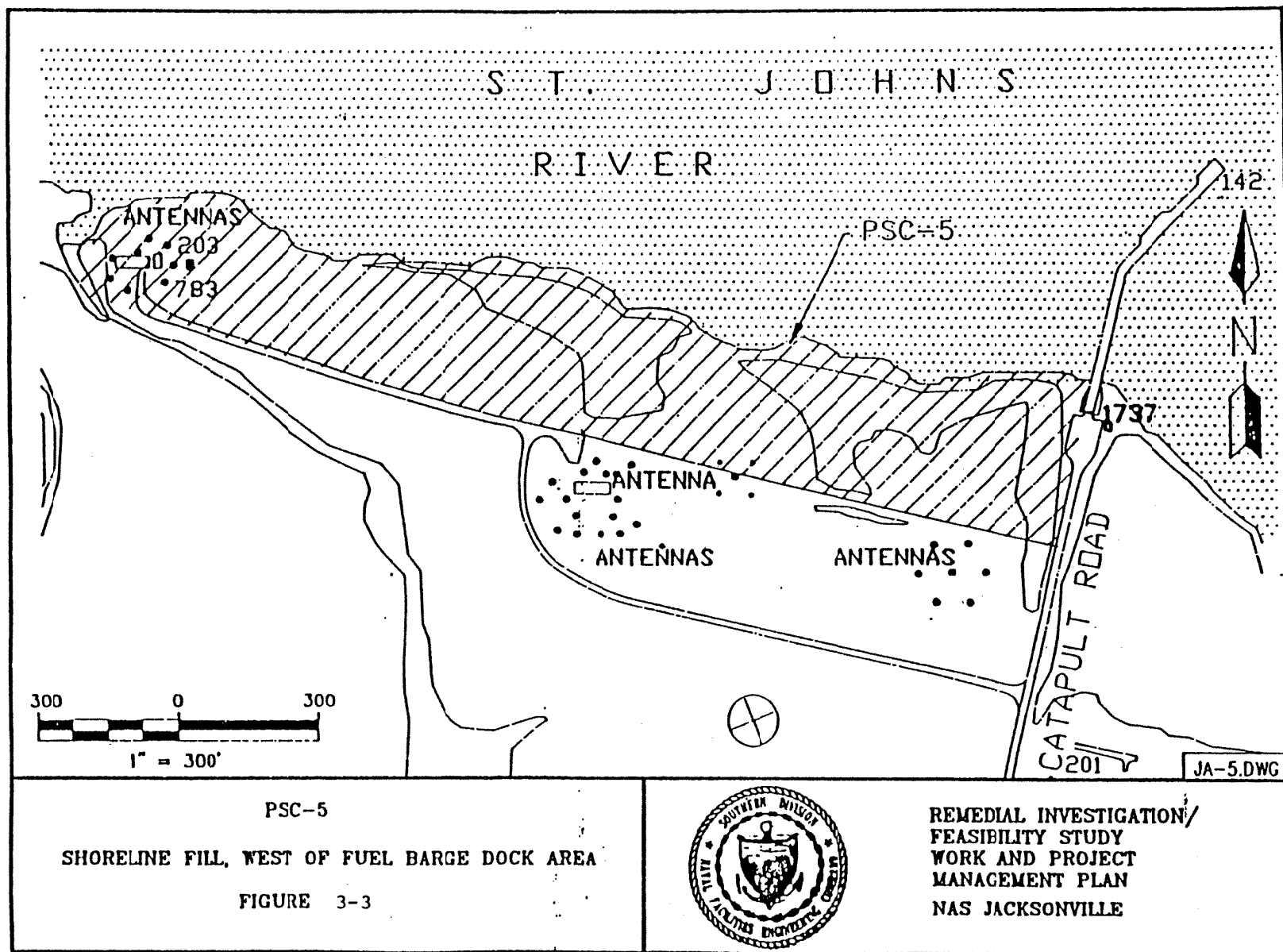
PSC No. 9: PSC No. 9 is an old disposal area, east of the fuel farm, located east of Catapult Road along the shoreline of the St. Johns River (Figure 3-5). This PSC contains construction debris, concrete rubble, and a few 55-gallon drums. BEI will conduct radiological surveys at PSC No. 9, according to protocols for a nonsuspect area, as defined in Section 5.

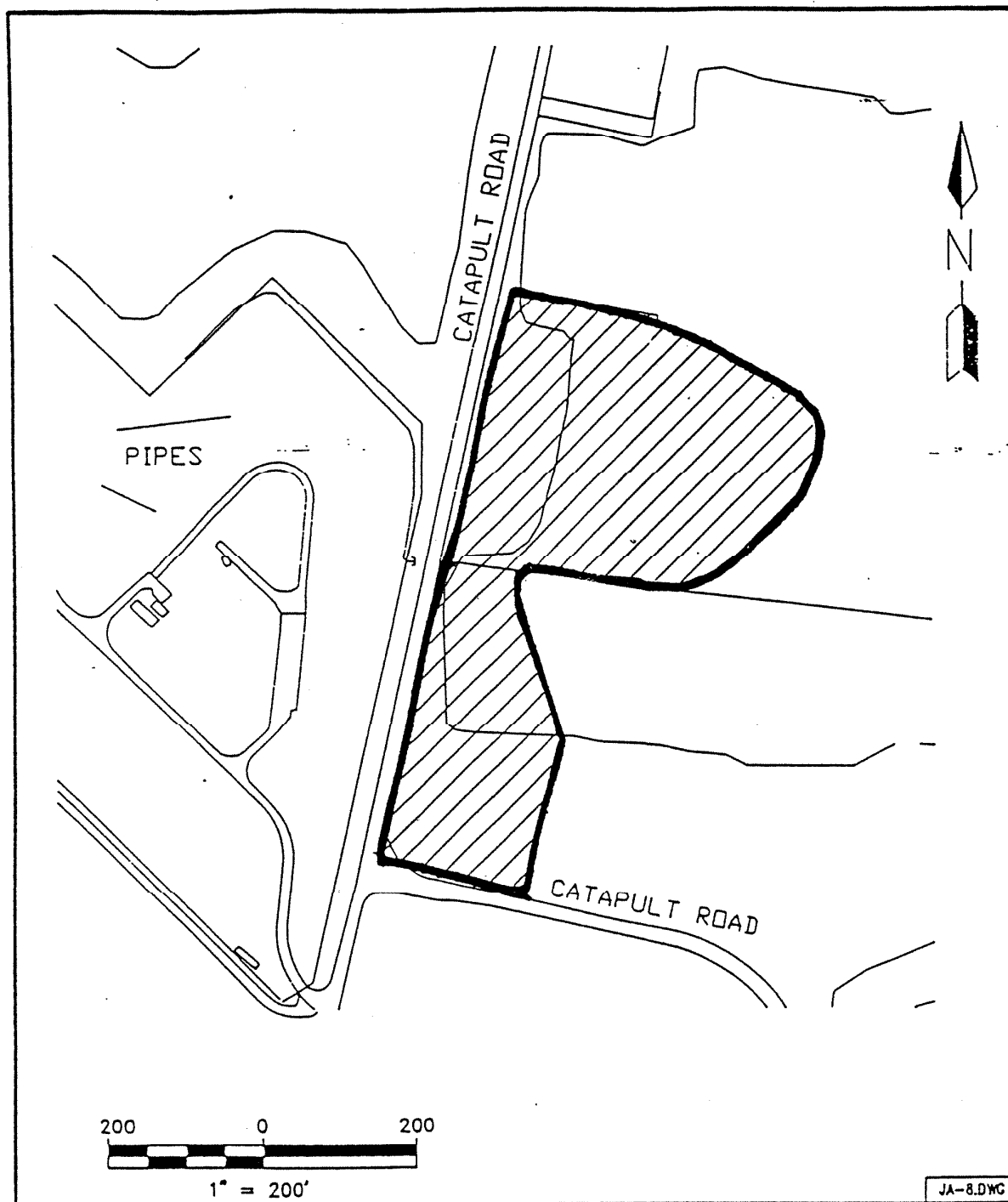
PSC No. 13: This PSC is the location of the radium paint disposal pit (Figure 3-6). The Navy used the area north of former Building 167 for the disposal of radioactive radium paint waste from aircraft instrument dial painting operations during World War II, continuing through the 1950s. The material was disposed into a burial pit approximately 40 feet by 50 feet and less than one foot deep. This PSC was excavated in the late 1950s, and the waste was reportedly moved to PSC No. 18 to the radioactive waste fill area. At the time of excavation, 3 to 5 millirem per hour above background were measured at the surface. BEI will conduct radiological surveys at PSC No. 13, including collecting both surface and subsurface soil samples to supplement a walkover survey as defined for a suspect area.

PSC No. 15: PSC No. 15 is the solvent and paint sludge disposal area, located south of the paint shop (Figure 3-7). This area was used for the disposal of solvents and paint sludges until 1978. BEI will conduct radiological surveys at PSC No. 15, according to protocols for a nonsuspect area, as defined in Section 5.

PSC No. 16: This PSC is the storm sewer discharge, located at what is referred to as Black Point (Figure 3-8). Various chemical wastes drained into the storm sewer system through cross connections within the sanitary sewer system over the years. The volume of material







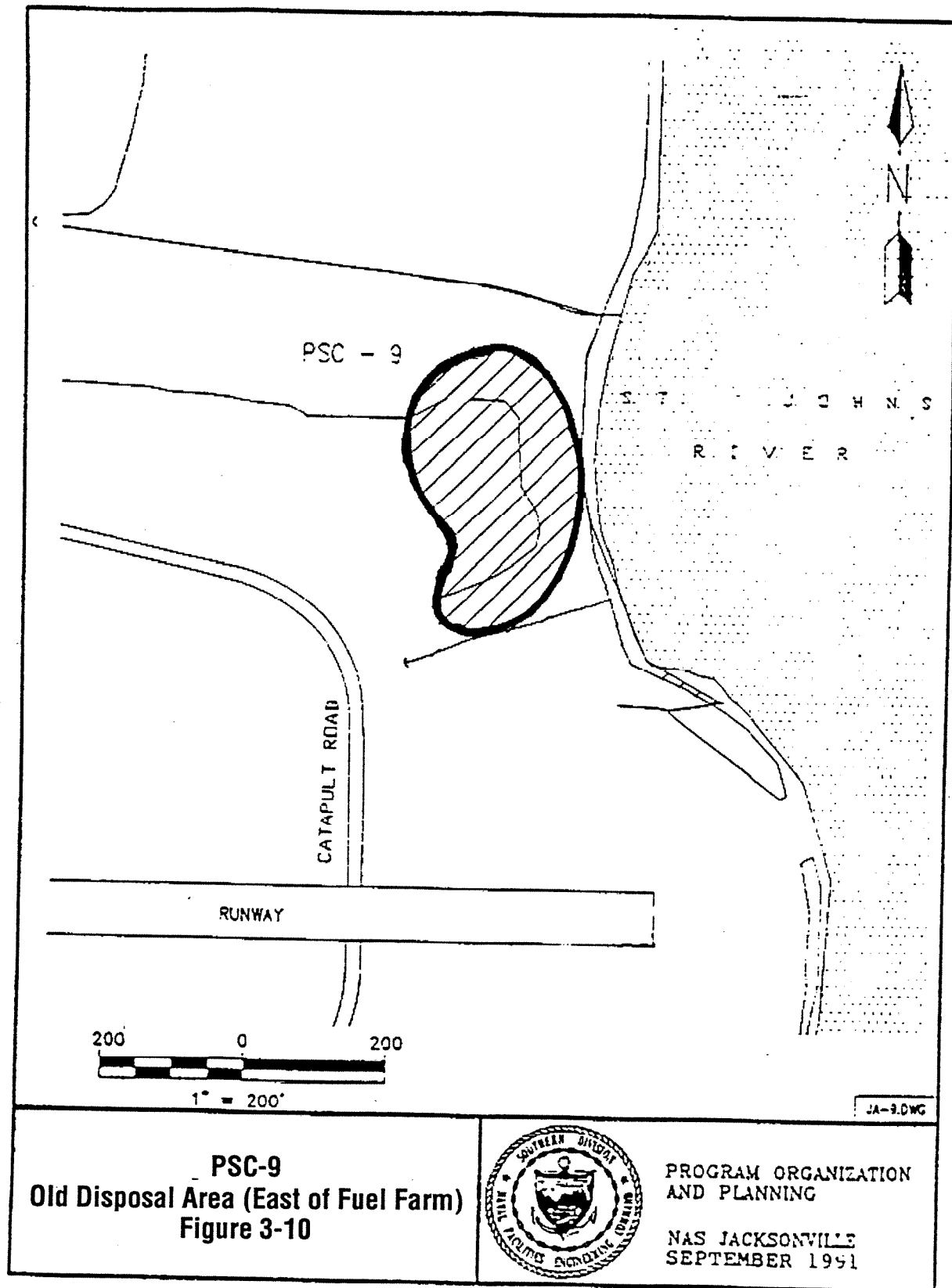
PSC - 8
Vacant Lot, Fuel Farm Area
Figure 3-9

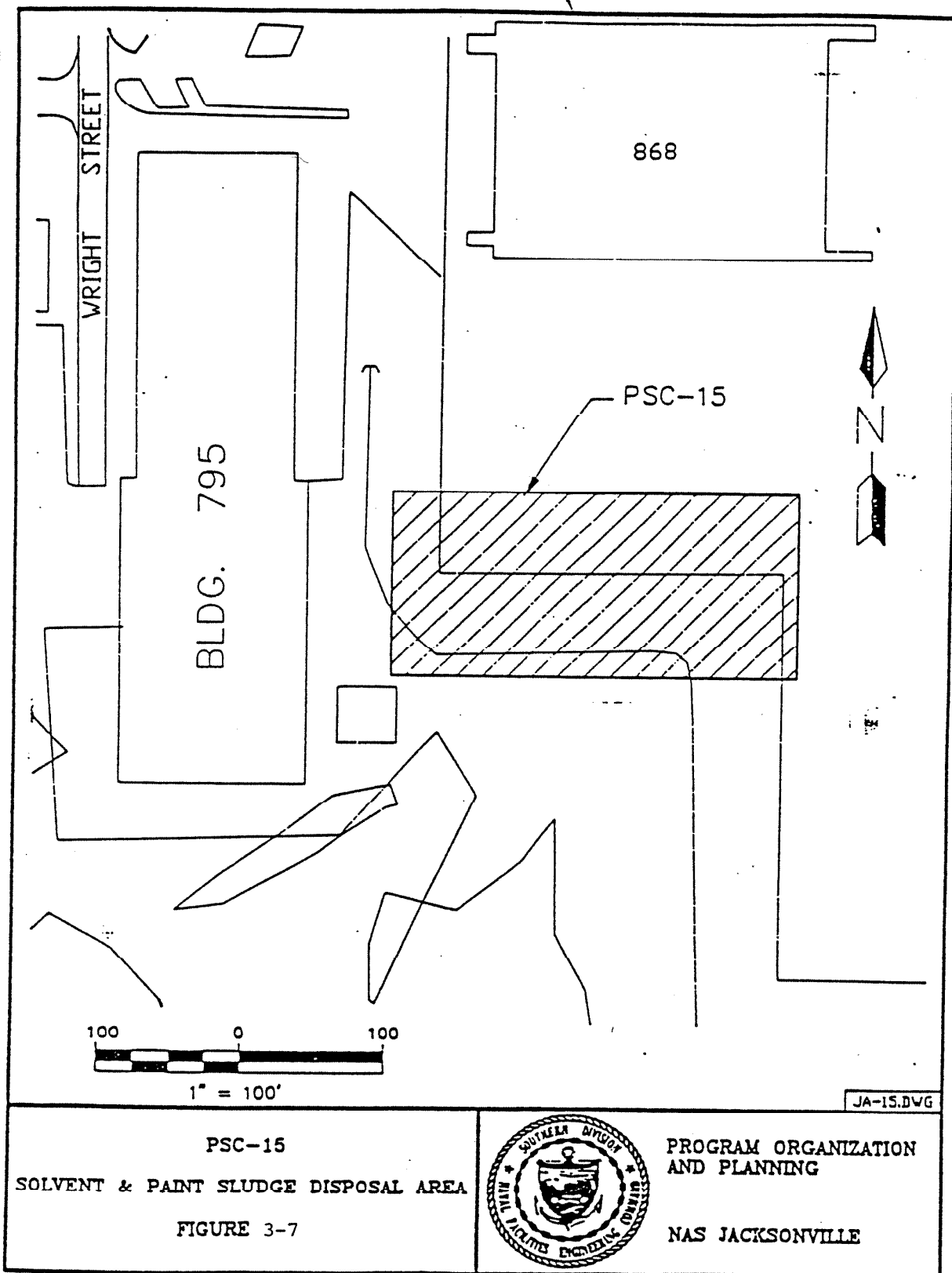


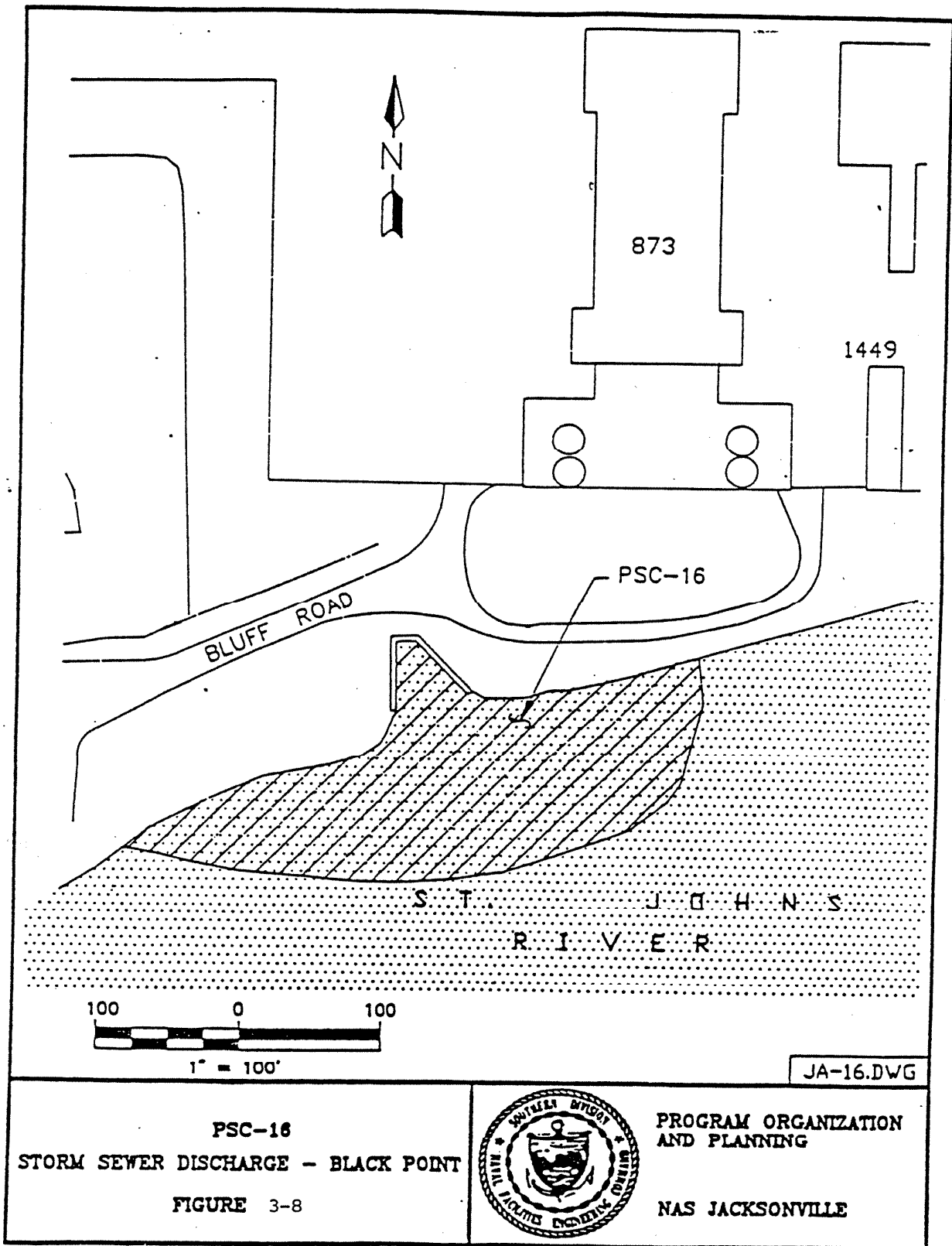
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deposited in this way has not been determined. BEI will conduct radiological surveys at PSC No. 16, in combination with PSC-5, 8, and 40, according to protocols for a nonsuspect area, as defined in Section 5.

PSC No. 17: PSC No. 17 is the glass bead disposal area, located in Mulberry Cove (Figure 3-9). This area was used by the Navy for the disposal of spent glass beads that were used in the abrasive blasting operation conducted at the base. Disposal began in 1965 and stopped in January 1981. Because this PSC is adjacent to and potentially contaminated by, PSC-18, a combined radiological survey will be performed. BEI will conduct radiological surveys at PSC No. 17 and 18, based on protocols for a suspect area, as defined in Section 5.

PSC No. 18: This PSC is a radioactive waste disposal area, located near the shoreline at Mulberry Cove (Figure 3-9). Radioactive radium paint wastes from aircraft instrument dial stripping operations were placed in a burial pit (PSC No. 13) until the late 1950s. When this operation ceased, PSC No. 13 was excavated, and the soil waste was disposed at PSC No. 18. Approximately 1500 cubic feet of contaminated soil is reported to have been disposed at PSC No. 18. The survey area for PSC 18 and PSC 17 extends 10 feet offshore into the river and approximately 50 feet north of Birmingham Road. BEI will perform radiological surveys at PSC No. 18, based on protocols for a suspect area, as defined in Section 5.

PSC No. 25: PSC No. 25 is the site of former Building 2038, naval hospital and ex-radioactive waste storage area. It is approximately 60 feet wide by 130 feet long. The area adjacent to former Building 2038 was used by the Navy for storage of hospital waste containing radioactive iodine (I-25), and other radioisotopes (Figure 3-10). This PSC was cleaned by a contractor, surveyed, and determined to be free of radiation. Building 2038 has subsequently been demolished. BEI will conduct confirmation radiological surveys at PSC No. 25, based on protocols for a nonsuspect area, as defined in Section 5.

PSC No. 26: PSC No. 26 is the old main registered disposal area (Figure 3-11). This area was used for the disposal of a variety of wastes, including spent solvents and spent oils. The Navy burned demolition and construction debris and solid wastes at the edge of trenches and pits, then bulldozed the remains into the trenches and pits and covered them. These operations began in 1940 and continued until 1968. Air pollution controls ended the burning, and the solid wastes were hauled offsite by a contractor. This PSC was officially closed on January 15, 1979.

A portion of PSC No. 26 was used for the disposal of low level radioactive wastes during World War II and until 1955. It is suspected that instrument dials and other low level radioactive wastes were disposed here. This area has been decontaminated by a contractor. BEI will conduct radiological surveys at PSC No. 26, in accordance with plans and protocols, described in Section 4 and Section 5.

PSC No. 29: PSC No. 29 is the organic disposal area (Figure 3-12). This area was used by the Navy for the disposal of organic debris (i.e., wood, grass clippings, etc.); however, there is evidence of other materials being disposed in this area. Crushed drums, construction debris, and scrap metal have been discovered at PSC No. 29. This area may also have been used as a borrow pit. BEI will conduct radiological surveys at PSC No. 29, based on protocols for a nonsuspect area, as defined in Section 5.

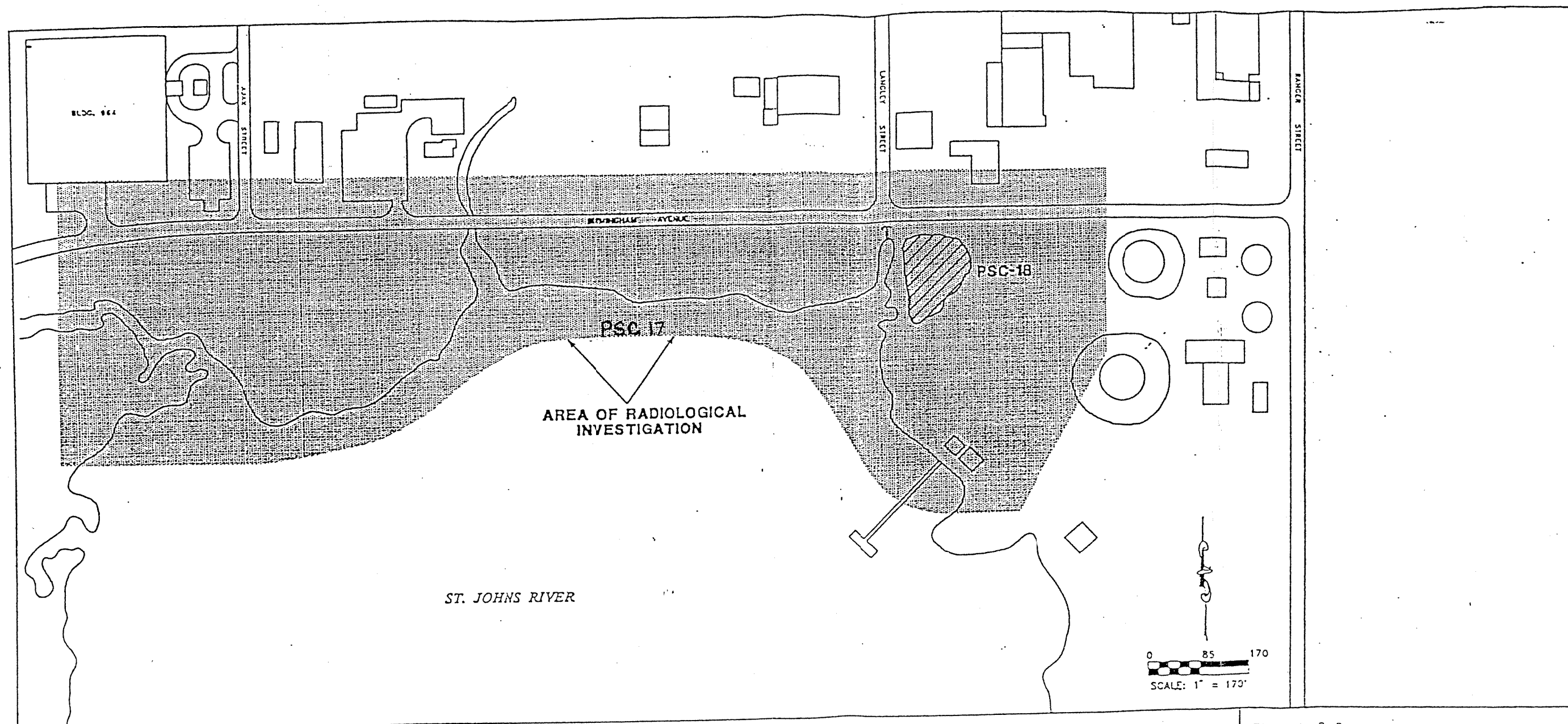
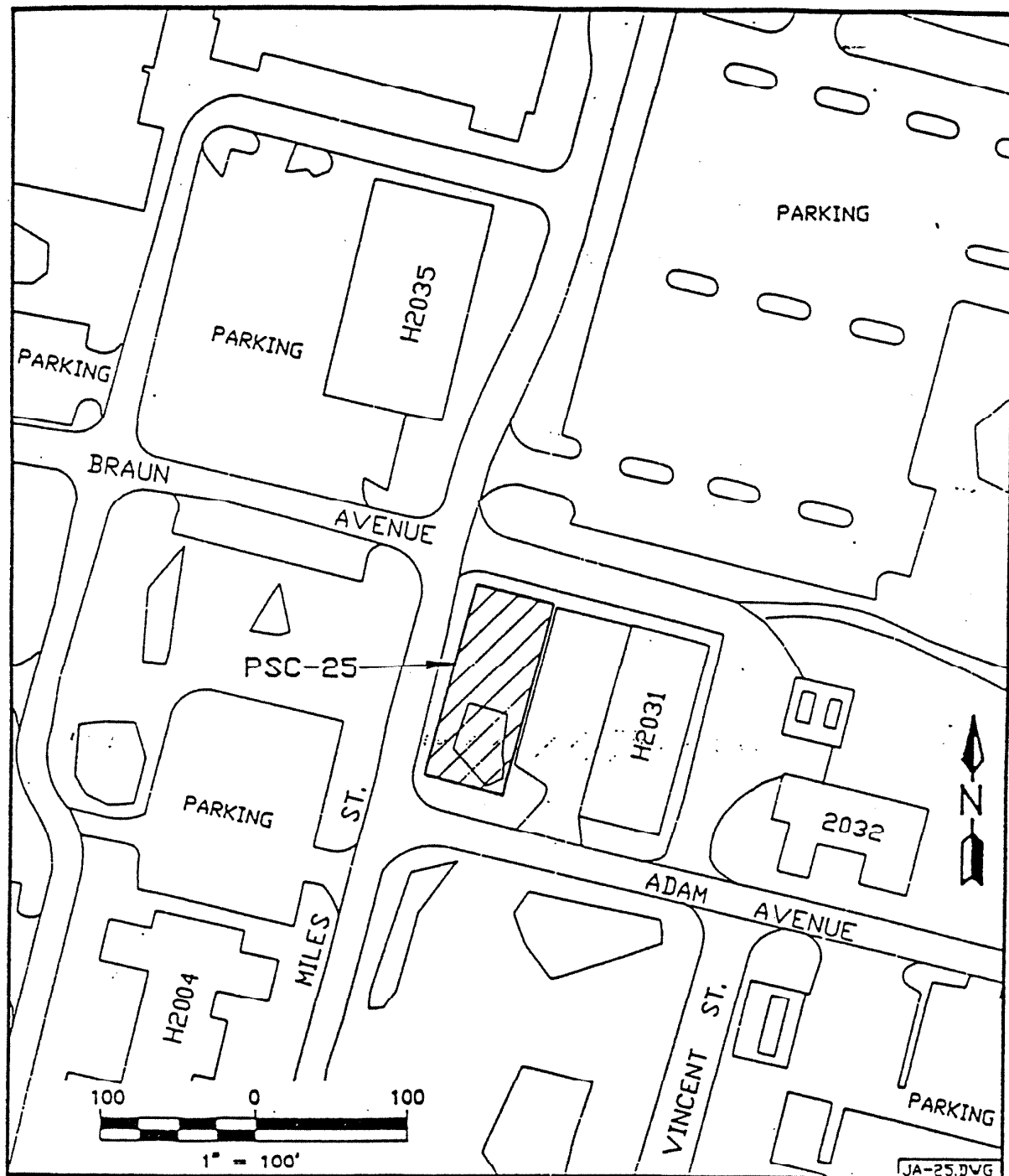


FIGURE 3-9
AREA OF RADIOLOGICAL
INVESTIGATION AT PSC-17 AND PSC-18



AREAS OF RADIOLOGICAL
INVESTIGATION

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PSC - 25
 FORMER BUILDING NO. 2038
 NAVAL HOSPITAL
 (EX-RADIOACTIVE WASTE STORAGE AREA)

FIGURE 3-10



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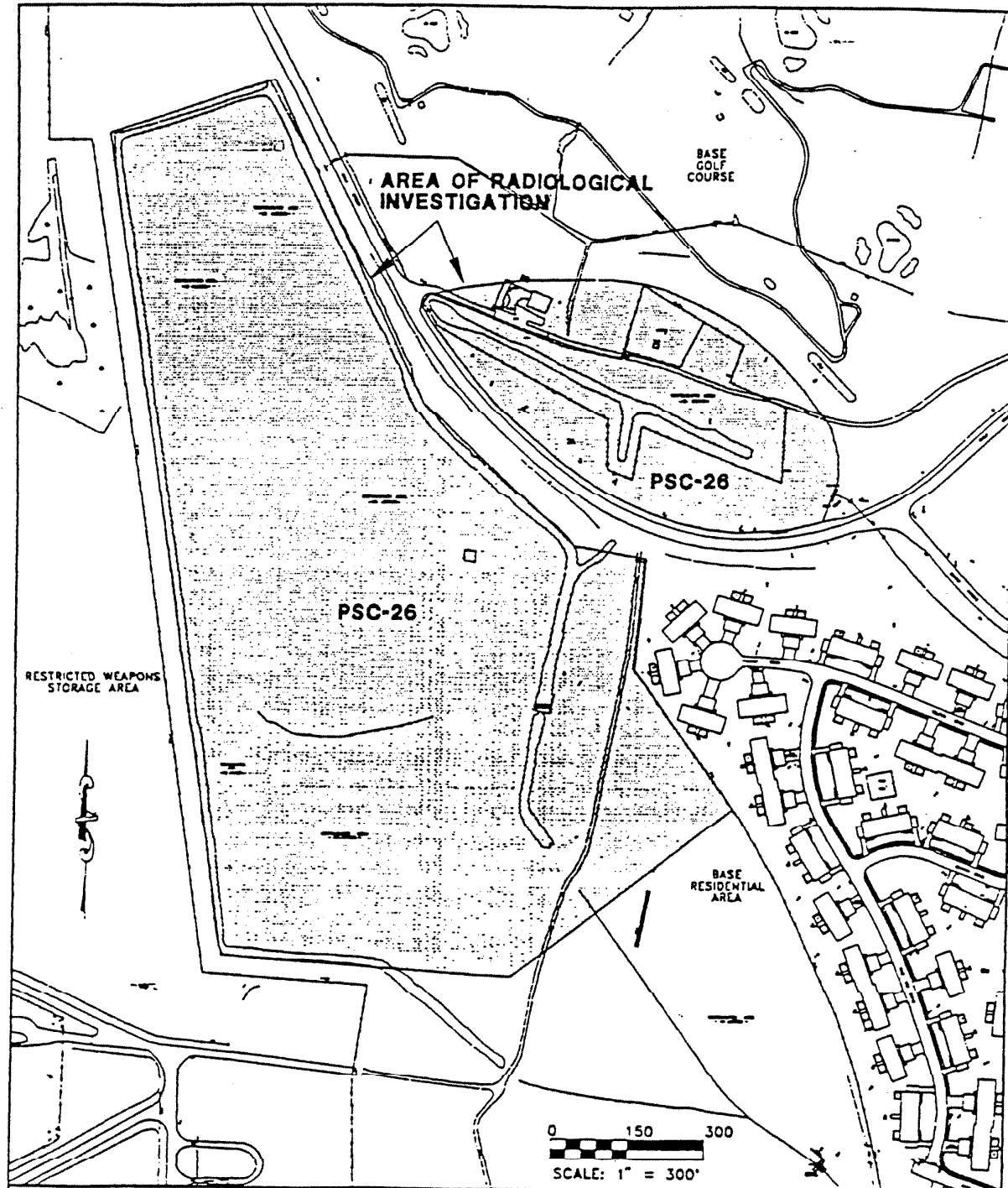
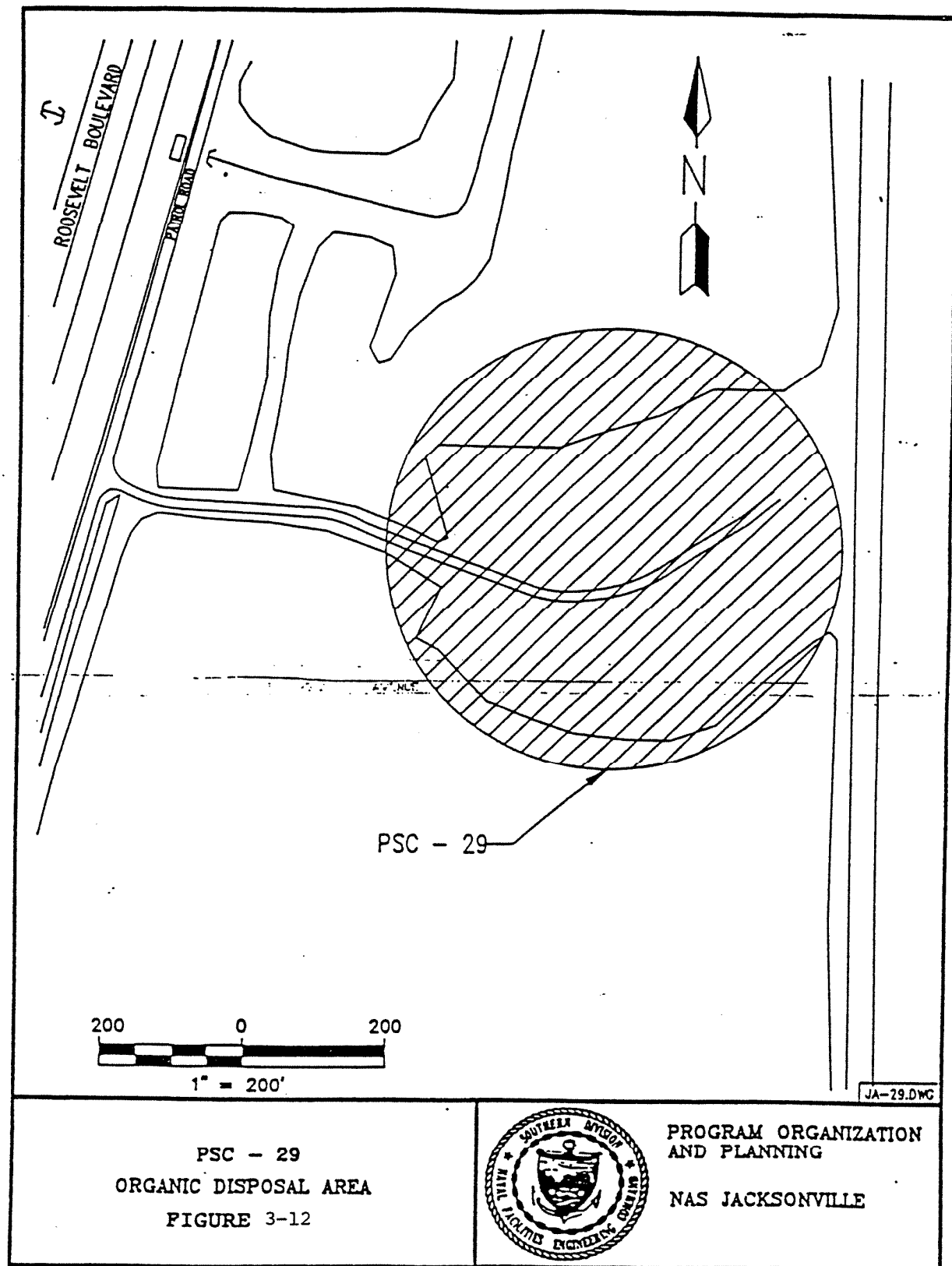


FIGURE 3-11
AREA OF RADIOLOGICAL
INVESTIGATION AT PSC-26



AREAS OF RADIOLOGICAL
INVESTIGATION

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JACKSONVILLE, FLORIDA



PSC No. 32: PSC No. 32 is the ex-base landfill, covering approximately 1.8 acres (Figure 3-13). During the 1960s, this landfill was used for the disposal of soil, refuse, construction debris, old vehicles, and household refuse. It is presently covered with tall grass and weeds. BEI will conduct radiological surveys at PSC No. 32, based on protocols for a nonsuspect area, as defined in Section 5.

PSC No. 40: PSC No. 40 is the ex-east wastewater treatment plant discharge area, located near the main runway (Figure 3-14). Primary treatment included settling for removal of solids and skimming for removal of oils and solvents. Secondary treatment was provided by trickling filter units. Effluent was discharged to the St. Johns River. This discharge resulted in a build-up of sediments in the cove east of runway 27; therefore, the Navy removed the east side plant from service in 1972, and the waste stream was diverted to the west side plant. Review of site operating history suggests that the "East" wastewater treatment system may have been contaminated by radium paint disposal into the drain system from aircraft instrument maintenance and repair. This could have potentially contaminated the outfalls into PSC-16 and 40. Additionally, dredge spoils from PSC-40 were placed in PSC-5, and 8. These four PSCs will be characterized as a single source term. PSC No. 40 extends 200 feet from the outfall along each bank and 300 feet into the St. John's River.

BEI will conduct radiological surveys at PSC No. 40, in combination with PSC-5, 8, and 16, in accordance with protocols for a nonsuspect area, as defined in Section 5.

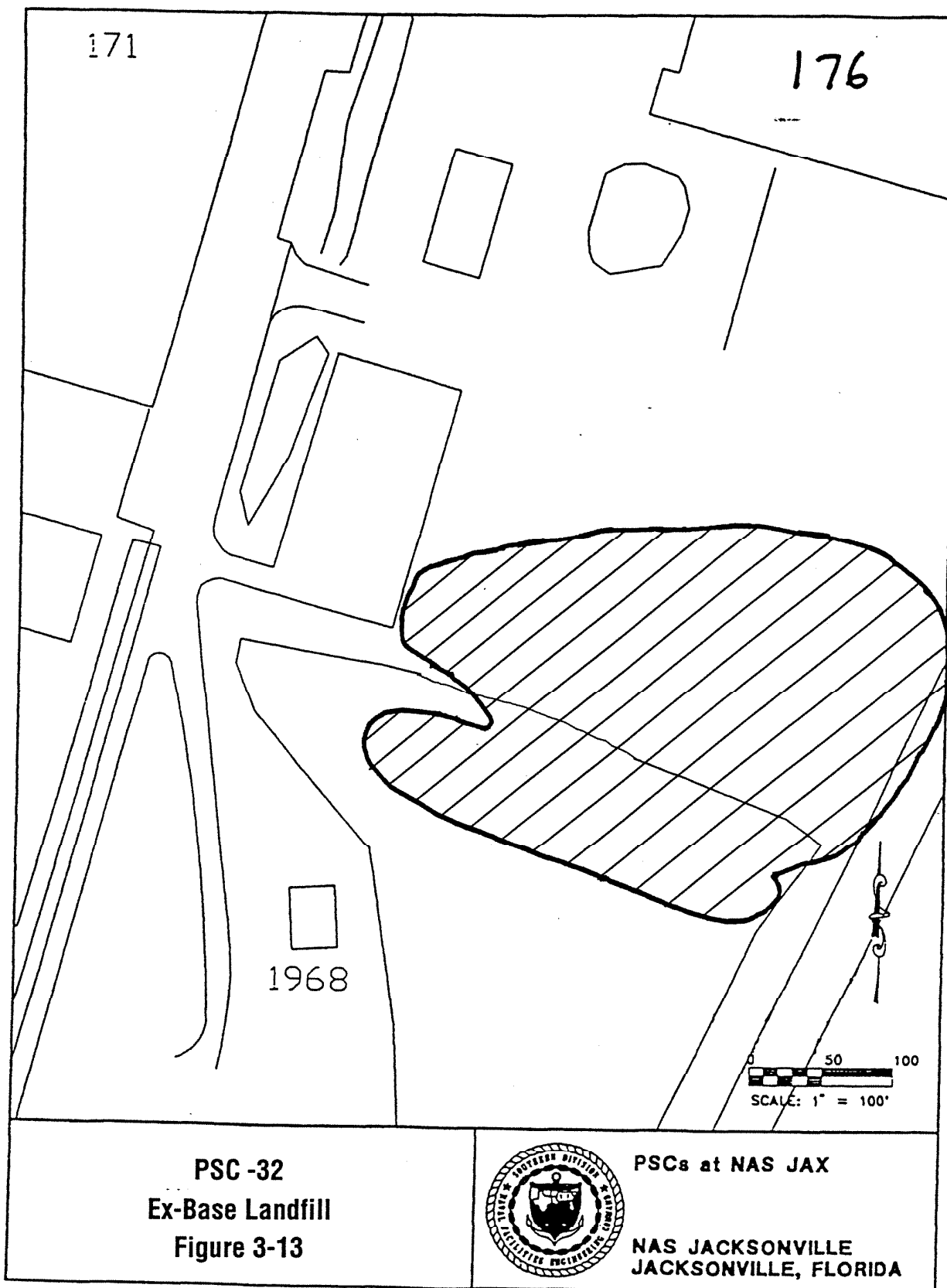
PSC No. 41: This PSC is the domestic waste sludge drying beds (Figure 3-15). The Environmental Protection Agency (EPA) classified the domestic sludge drying beds as surface impoundments operated to treat Resource Conservation and Recovery Act (RCRA) F006 and F019 listed hazardous wastes. An average of 90 gallons per day of sludge was dewatered in domestic sludge drying beds during the operation.

PSC No. 41 covers an area of approximately 50 feet wide by 250 feet long. BEI will conduct radiological surveys at PSC No. 41, in combination with PSC 3, 4, 42, and 43.

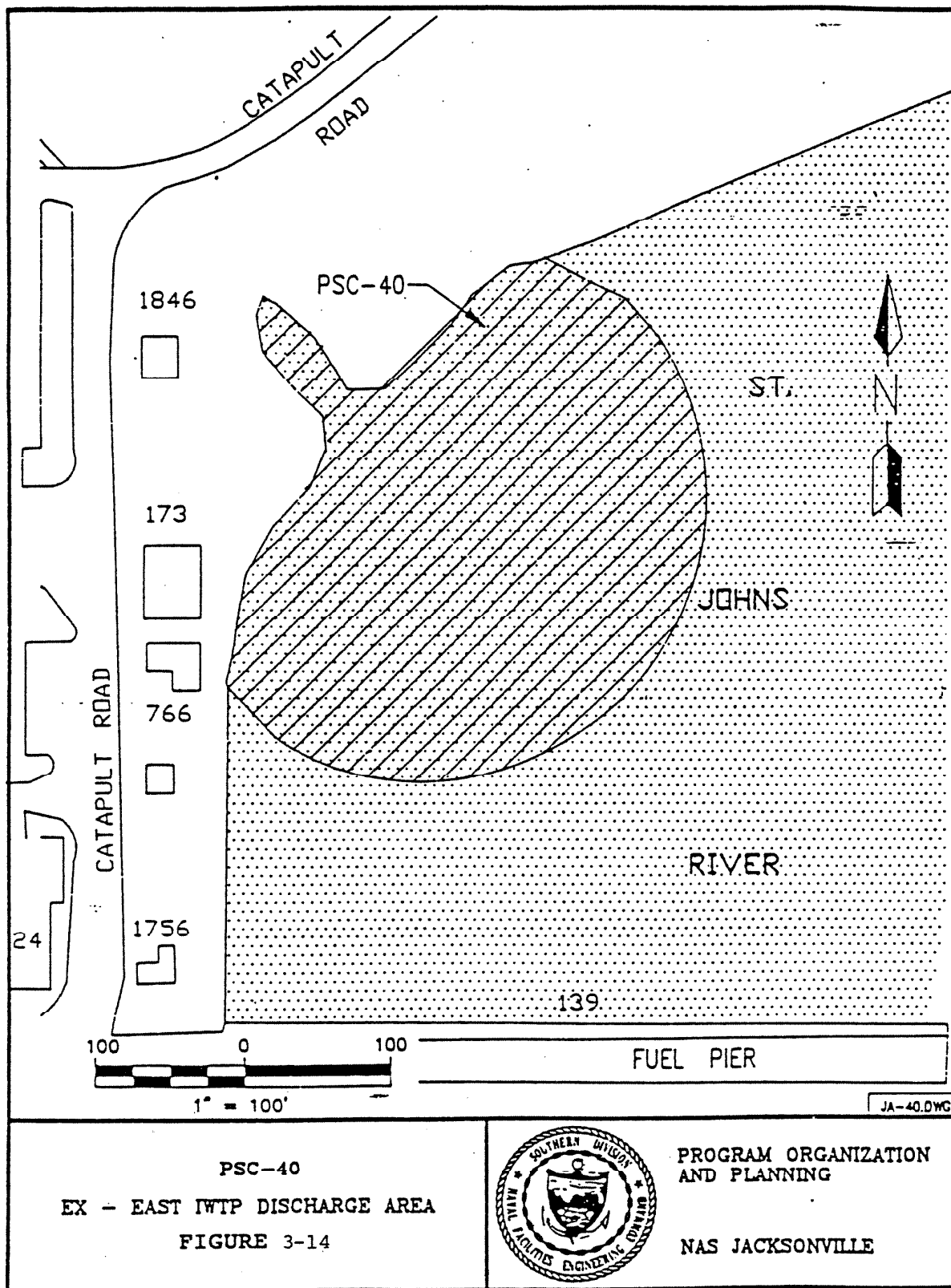
PSC No. 42: PSC No. 42 is a former polishing pond for the waste water treatment plant effluent (Figure 3-16). This polishing pond was classified by the EPA as a surface impoundment to treat RCRA F006 and F019 listed hazardous wastes. The polishing pond received 2.3 million gallons per day of treated effluent from the wastewater treatment plant for final clarification before chlorination and discharge to the St. Johns River. PSC No. 42 covers an area approximately 400 feet wide by 500 feet long.

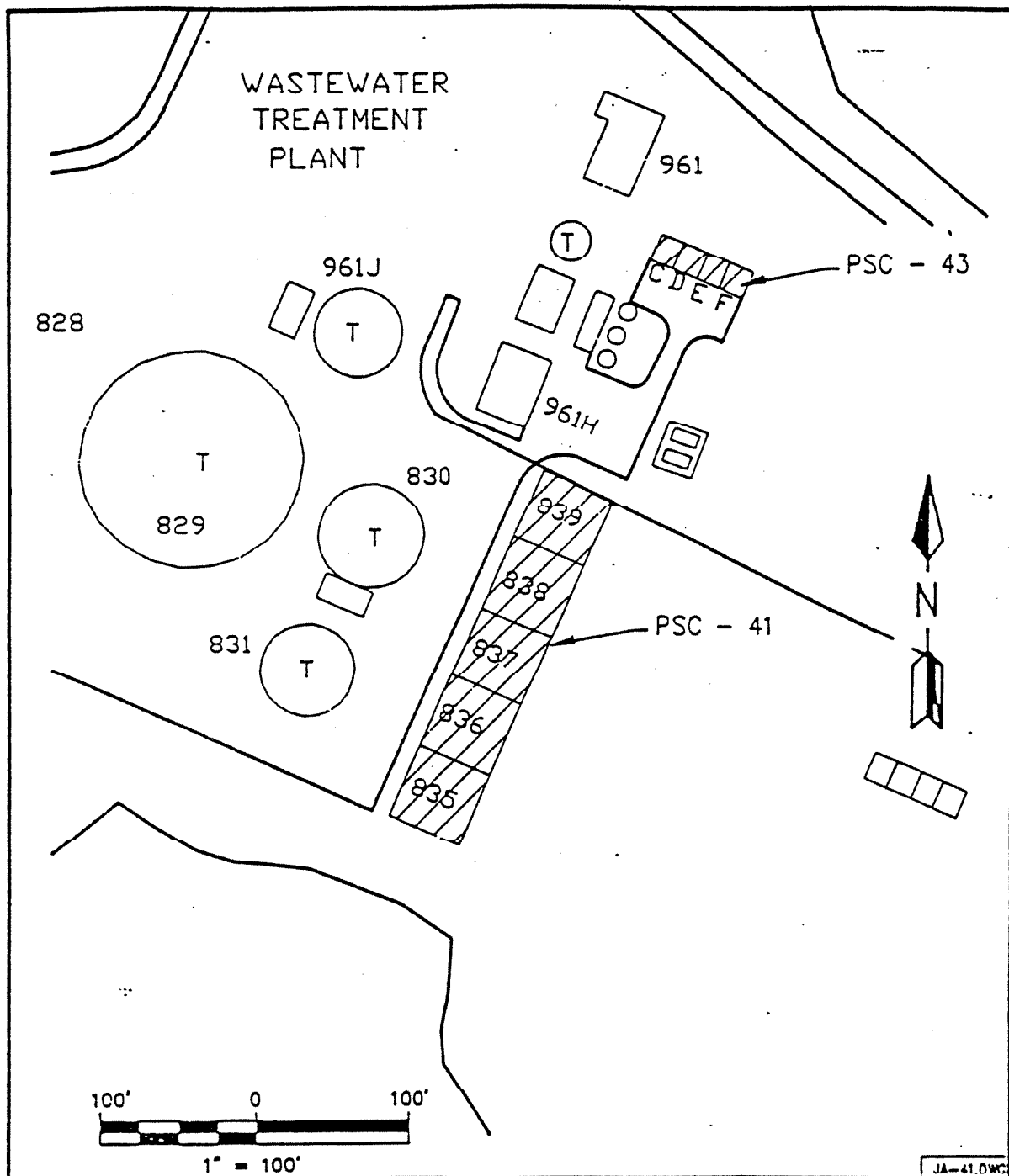
BEI will conduct radiological surveys at PSC No. 42, in combination with PSC 3, 4, 41, and 43.

PSC No. 43: This PSC consists of the industrial waste sludge drying beds, which were built in 1980 and operated to dewater metal hydroxide sludges from electroplating operations (Figure 3-15). Approximately 8,250 gallons of dried sludges were excavated and removed from the surface impoundment annually. The sludges may have contained heavy metals and organic solvents. PSC No. 43 covers an area of approximately 25 feet wide by 25 feet long.



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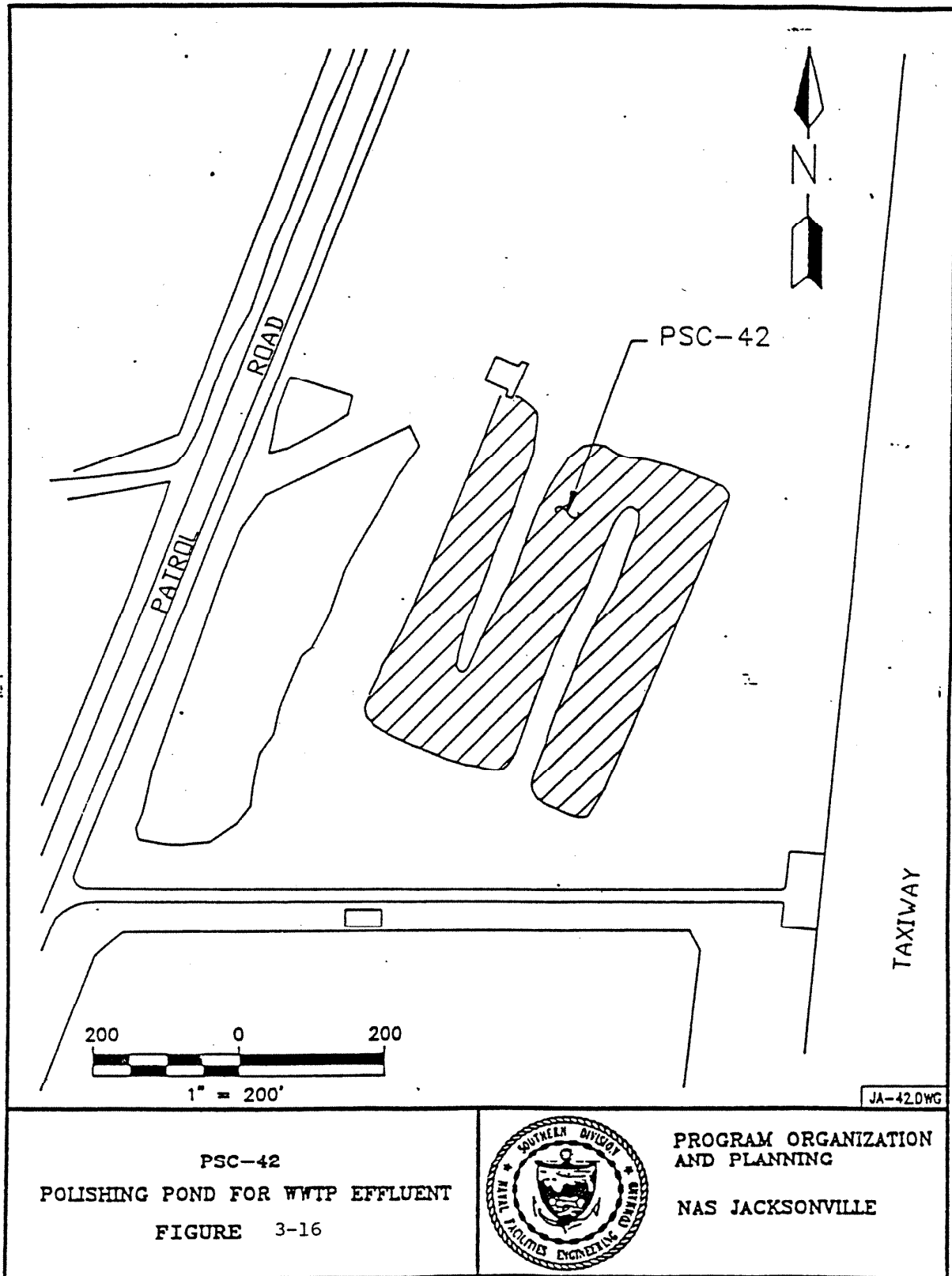




PCS-41: DOMESTIC WASTE SLUDGE
DRYING BEDS
PCS-43: INDUSTRIAL WASTE SLUDGE
DRYING BEDS
FIGURE 3-15



PROGRAM ORGANIZATION
AND PLANNING
NAS JACKSONVILLE



In 1988, the industrial waste sludge drying beds were taken out of service and all sludge was removed. The underlying drainage sand and filter underdrains were left in place. BEI will perform radiological surveys at PSC No. 43, in combination with PSC 3, 4, 41, and 42.

Building 164: Building 164 is located in the southwest quadrant of NAS Jacksonville. It is a one story warehouse building measuring approximately 200 feet by 497 feet. Radiological contamination may be present on the floor surface as a result of possible spills or leakage of stored radioactive materials. BEI will perform a radiological survey of the building's concrete floor. All areas of the floor surface are accessible.

4.0 SCOPE OF WORK

The remedial action objective at NAS Jacksonville is to survey and remediate 18 PSCs, as necessary, to protect public health and the environment. To accomplish this objective, the following services will be performed:

- mobilization
- clearing
- civil surveying
- radiological surveying
- radiological remediation
- waste management
- safety and health
- quality control

The following subsections provide descriptions of each of these components of the RRWP.

4.1 MOBILIZATION

Mobilization will include delivering to the jobsite and work areas all construction equipment, tools, materials, supplies, and miscellaneous articles and establishing a work force sufficient to commence and sustain construction activities as required.

As part of mobilization, BEI will establish two on-site trailers consisting of a personnel decontamination trailer, as well as a personnel office trailer. Setup of these trailers will include electrical, sewer, and water connections. In addition, BEI will construct an area for temporary placement of two 5,000 gallon bladder tanks for containment of decontamination water or other liquid waste as required. The design and installation of the bladder tanks will provide secondary containment. If necessary, a temporary decontamination pad will be constructed in an area near the bladder tanks.

4.2 CLEARING

Areas to be radiologically surveyed and potentially remediated shall be cleared of all debris and vegetation as necessary in order to provide access for the radiological walkover survey. Clearing shall consist of removing all designated vegetation and debris within the limits of areas to be surveyed. Grass within the limits of clearing shall be mowed to a maximum height of two inches. Clearing shall include the removal of trees and shrubs with a trunk diameter less than

two inches. Trees and shrubs with trunk diameter greater than two inches and vegetation along shorelines shall not be removed until BEI has coordinated removal with the NAS Jacksonville environmental officer. All removed above-ground materials shall be suitably reduced in size for safe transport and storage. All above-ground cleared materials generated as a result of clearing operations shall be stockpiled in areas as directed by the Navy. The disposition of all materials generated as a result of clearing operations shall be determined in the field.

4.3 CIVIL SURVEYING

A grid will be established by a civil surveyor for conducting the gamma walkover surveys. The grid will be correlated with the Florida State plane grid system. Civil surveying for PSC 26 will be completed by ABB-ES; BEI will complete civil surveying for the remainder of the PSCs in this scope of work. Civil surveying will be performed in accordance with the Technical Specification for Civil Surveying, included in Appendix A.

4.4 RADIOLOGICAL SURVEYING

4.4.1 Characterization

Based upon operating history and preliminary radiological measurements performed by the Navy, the 18 PSCs have been designated as either a suspect or nonsuspect area. Radiological measurement protocols have been defined for each designation in Section 5. In addition, remediation of PSC will be followed by a verification survey that is based upon protocols similar to those defined for a nonsuspect area. Adjacent PSCs that may be characterized radiologically by the same source term have been combined for the purposes of radiological characterization.

The following PSCs identified as "suspect areas" will have a radiological walkover survey performed on 100 percent of the area to locate sources near the surface and other areas of elevated gamma-emitting contamination (hot spots):

PSCs 13, 17, 18, and 26

Based upon the gamma walkover surveys in PSC 13 and 17/18, contaminated soil will be excavated and placed in containers for final disposition by the Navy. At the completion of excavation activities, a final verification survey will be conducted. A composite soil sample for each area will be collected for TCLP analysis to characterize the waste.

In PSC 26, any area of elevated gamma activity will be visually inspected to determine if the activity is associated with a discrete device. If a device is identified, it will be collected and packaged for disposal by the Navy. A sample of soil, beneath the location of the device will be collected. At the completion of these surface activities, the samples collected will be composited for a TCLP analyses to assist in waste characterization.

Building 164, used to store containerized radioactive material, is cleared of all materials and equipment. The building will be treated as a nonsuspect building and a verification survey will be conducted to confirm the absence of any residual radioactive material. This will be accomplished by performing scan surveys using an alpha and beta detector to indicate the presence of elevated or "hot spot" activity.

The following PSCs have been designated as "nonsuspect" and will have a radiological walkover survey performed on grid lines to confirm the absence of gamma-emitting contamination:

PSCs 3/4/41/42/43, 5/8/16/40, 9, 15, 25, 29, and 32.

A survey grid will be established on 10-meter increments. A scan survey will be conducted along the grid lines. If elevated areas of radioactivity are detected, then soil sampling on the surface at these locations will occur following the same protocol as for PSCs 13, 18, and 26. If radioactive contamination is not detected during the walkover survey, confirmation soil sampling will be conducted as described in Section 5 of this Work Plan.

Sediment samples will be collected from outfall locations (and the moat surrounding PSC 26) from the following PSCs:

PSCs 16, 17, 26, 40, and 42

Sediment sampling will be conducted in the St. Johns River adjacent to PSC 5 should radioactive contamination be detected on PSC 5, extending to the shoreline/water interface.

All samples collected for analysis will be analyzed by gamma spectroscopy at an offsite laboratory.

4.4.2 Contamination Control and Field Support

BEI will provide radiological and contamination control support to all field activities involving potential radioactive material handling based upon the results of the characterization described in Section 4.4.1. Areas of elevated activity will be identified and entry/egress from those areas will be controlled. Contamination control and monitoring equipment and protocols appropriate to the levels and types of contaminants ascertained from characterization.

Monitoring will be provided on all materials produced by subsurface characterization activities. In addition, BEI will perform radiological surveys and decontamination, as appropriate before release, on all equipment used in a radiological controlled area. The results of these radiological surveys will be documented. Drums being filled with contaminated soil and other radioactive contaminated materials will have the dose rate assessed on them, and the material with which they are filled will be sampled and analyzed.

4.5 RADIOLOGICAL REMEDIATION

Removal of radioactive contamination will be conducted to the depth detected in all PSCs except PSC 26. This will coincide with the radiological walkover surveys. In PSC 26, only discrete objects, indicating radiological contamination, will be removed. The contaminated material will be removed by using hand shovels or similar tools and placed in 55-gallon drums. One composite sample will be obtained for each waste type, to be analyzed for TCLP.

On all PSCs where radioactive contamination is identified, both the horizontal and vertical boundaries of contamination will be established at the location where contamination is detected

using field survey instruments and analytical data. These boundaries will be recorded in field logs and excavation of the contaminated material will be conducted.

4.6 WASTE MANAGEMENT

Hazardous waste will be managed as directed by the Navy Public Works Division. The Hazardous Waste Management Plan for the Naval Facilities Engineering Command, Southern Division, will be used as guidance for all hazardous waste management activities including radioactive materials handling at NAS Jacksonville. Accountability for waste is required from the time of containerization until turnover to the Public Works Department of NAS Jacksonville. A waste inventory will be maintained, packaged waste will be marked with labels, and relevant data entered into the data collection system.

The Hazardous Waste Management Plan establishes guidelines and minimum programmatic requirements by which all wastes will be managed.

4.7 SAFETY AND HEALTH

The NAS Jacksonville Site Specific Health and Safety Plan will be followed during radiological remediation of the site.

4.8 QUALITY CONTROL

The NAS Jacksonville Site Specific Quality Control Plan will be followed during radiological remediation of the site.

5.0 SAMPLING AND ANALYSIS PLAN

Radiological surveys of NAS Jacksonville indicated four suspect areas consisting of four PSCs with possible residual activity above background levels attributable to prior operations at the base. Activity in three of the PSCs are defined as "hot spots" that would be characteristic of radium-226 aircraft instrument dials; the fourth PSC contains more uniform contamination that may be the result of disposal of sand-blasting grit containing elevated levels of thorium (radium-228). Radiological remediation will be performed in these areas, as appropriate, and supplemental radiological characterization will be performed to support ABB-ES's completion of the RI.

5.1 RADIOLOGICAL SUPPORT - SUSPECT AREAS

The suspect areas are identified as PSCs 13, 17, 18, and 26. The first priority for radiological remedial action is PSC No. 26 and PSC No. 13. PSCs 17 and 18 will be surveyed, and remediated if required, as a second priority. The radiological support (i.e., surveying and remediation) to be provided in the suspect areas will be comprised of the following primary activities:

- surface measurements
- surface remediation

- subsurface sampling, as required
- sediment sampling and analysis

5.1.1 Surface Measurements

For those PSCs that have been designated as suspect, the following survey protocol will be used:

- (1) Complete clearing of underbrush on the PSC. The PSC is to be measured into square grids roughly 10 meters on a side. This will assist the data acquisition efforts in assuring that all areas are covered by defining shorter straight-line distances to travel, and it makes data acquisition and record keeping more manageable.
- (2) Survey a path approximately one meter wide using a gamma scintillation detector, (e.g., 2" x 2" NaI), with ratemeter and audio output, at a foot speed of approximately 2 ft/sec.
- (3) Survey the entire surface by north-south traversing at one meter intervals between the traverse path, using a two-man survey team.
- (4) Note in the field logs each location that indicates an elevated audio signal. The radiological technician should maintain traverse speed, while the support technician marks (flags or paints on the ground) the location responsible for the elevated reading.
- (5) At the completion of the 10 m x 10 m grid survey, return to the marked locations. Using the same detector, but with a scaler, note the level of activity and the extent (size and shape) of the activity above background. Record this information using field survey logs.
- (6) Repeat for each 10 m x 10 m grid until the entire PSC is surveyed.
- (7) Evaluate the results to identify those PSC areas with activity levels in excess of two times background and initiate remediation planning.

The result of performing steps (1) through (7) above, for the entire areas of PSCs 13, 18, and 26 is a 100 percent gamma walkover scan.

For Building 164, a 100 percent alpha and beta scan will be performed. A survey grid will not be established in this building by the civil surveyor; however, the radiological technicians may measure off areas to allow for assurance that all the floor surface is covered during the survey. Any elevated areas will be marked by the support technician. All marked locations will be returned to, and a fixed position survey will be conducted to determine the level of activity and extent of the activity above background.

5.1.2 Remediation

Based upon the results obtained in 5.1.1, initiate remediation activities. Assemble the following items:

- shovels
- dig bars (or picks)

- 17H (55-gallon) drums
- sheet plastic
- personal protective equipment (PPE) requirements based on results from 5.1.1
- portable alpha meter
- portable beta/gamma meter
- gamma detector used in 5.1.1
- soil sample containers
- plastic bags

For areas exceeding 5 Pci/gm or 100 dpm/100 cm² perform remediation as follows:

- (1) Place a drum on the plastic sheeting and excavate the contaminated soil into the drum. During removal of the contaminated soil, an effort should be made to remove and segregate any solid devices containing radioactive material. These devices or pieces of devices should be placed in a plastic bag for separate handling, later to be stored in a 17H drum. The soil excavation will be guided by using field instrumentation to detect contaminated material for containerization. In addition, dust control measures will be implemented as required.
- (2) Obtain activity levels representative of the radioactive materials (i.e., radium-226) in the drum and of metals, pesticides, herbicides, volatile organics, and semi-volatile organics by collecting approximately 400 grams of soil as the drum is filled, at approximately 20 percent increments for a total of five 400 gram samples. The five samples will be composited, resulting in approximately two 1 kg samples for each drum. One composited sample will be containerized for sample preparation and analysis offsite at the radiological laboratory.

The other representative sample from each drum will be composited to prepare 1-3 samples per site and sent to an offsite laboratory for toxicity characteristic leaching procedure (TCLP) analysis. Sample mixing and dividing, containerization, preservation, and holding times will be accomplished per Sections 4.3 and 4.4 of the FDER SOPs (DER-QA-001/92). Decontamination of the sample mixing bowls and other sampling equipment will be performed in accordance with Section 4.1 of DER-QA0-001/92.

- (3) Continue the excavation until the excavated site is less than two times background on the gamma detector. The use of two times background is a screening criterion for fixed position surveys; post-remedial action measurement criteria will be based on sample analysis and factors established for meters correlating counts per minute to Pci/g. Obtain a 1 kg soil sample from the excavated area for offsite analysis. Obtain a beta/gamma and alpha survey reading in each excavated area. Record the results.
- (4) Continue to the next identified (i.e., marked) area and repeat until the entire PSC is remediated or the survey of elevated surface activity is recorded.
- (5) Perform offsite analyses for radium-226 on the soil samples. Radium-226 and radium-228 analyses will be conducted on sediment samples collected from PSC No. 17 due to disposal of spent glass beads used in abrasive blasting operations. Follow up remediation activities may be required based on the sample results.

- (6) Perform gamma exposure rate measurements 3 ft above the ground surface, using a pressurized ionization chamber (PIC). The PIC measures a response to gamma radiation that is proportional to exposure in roentgens. PIC measurements will be made approximately five per acre but no less than one per site, after excavation of contaminated material. Gamma exposure rate measurements will be made to support evaluations of human health from residual radioactivity.
- (7) Prior to leaving the PSC, remove all PPE and place in plastic bags. Perform a whole body frisk, with emphasis on hands, feet, and face areas, using both the beta/gamma detector and the alpha detector. Record results, even if no activity is noted.
- (8) In Building 164, areas in excess of two times background will be cleaned using wiping cloths and/or wire brushes and a small amount of detergent until the activity level on the floor surface decreases to acceptable levels. If this decontamination technique does not lower the floor surface activity level, more aggressive, abrasive techniques will be employed.

Two PIC measurements will be made in Building 164 upon completion of the remedial action to measure gamma exposure rates. If remediation is required in Building 164, a final survey will be performed based upon fixed alpha and beta measurements on 1m × 1m grids.

5.1.3 Sediment Sampling

Sediment sampling and analysis will be conducted in those suspect PSC areas that are not amenable to the walkover survey technique. This approach will be employed at PSCs 17 and 26 to evaluate shoreline and outfall areas. In addition, sediment sampling may be conducted in the St. John's River adjacent to PSC 5, if radioactive contamination is detected on PSC 5, and it extends to the shoreline/water interface. Grab sediment samples will be collected using a clam shell, mounted to an outrigger on a pontoon boat at PSC No. 17. The location where the sediment sample is collected will be determined with an electronic distance meter. Coordinates at two locations on shore will be established, and the distance meter will be used to measure the distance from each of these two locations. At PSC No. 26, sediment samples will be collected with the clam shell, attached to a boom that is mounted to the backhoe; or the backhoe will remove some sediment, the technician will obtain the sample from the backhoe bucket, and the contents of the bucket emptied back into the moat. The sampling apparatus and other items used in the radiological sample locations will be cleaned before work commences at the next sample location so that they are free of visible soil, debris, and other foreign matter.

Assemble the following materials:

- clamshell bucket
- sample containers
- sheet plastic
- PPE
- bucket/tray
- portable alpha meter
- portable beta/gamma meter

- rags
- distance meter

- (1) Obtain 5 randomly selected sediment samples from PSC No. 17. A pontoon boat will be used during the sampling. Note locations to extent practical and record in field log. Obtain approximately 1 kg of sediment (< 50% water) for sample preparation and analysis.
- (2) Sediment samples will be collected from the moat surrounding PSC No. 26 (7 random and 3 biased samples have been assumed).
- (3) Five (5) randomly selected sediment samples will be collected from PSC-16, 40, and 42. These samples will be collected using the same approach described for PSC-17.

Sediment samples will be analyzed for radium-226 by gamma spectroscopy at an offsite laboratory. Offsite analysis of radium-228 will also be performed for sediment samples collected from PSC No. 17.

5.1.4 Soil, Sediment, and Water Analyses

Soil samples and sediment samples collected from the PSCs will be prepared for analysis by drying and crushing (at a minimum to sieve through a #6 screen). The samples will be analyzed by gamma spectroscopy for radium-226, and in some instances, as noted in this Plan, for radium-228, in an offsite laboratory. In addition, radium-226, radium-228, and total uranium analyses will be performed for the background samples.

The composite samples collected from the drums as they are being filled with radioactively contaminated soil will be analyzed for radium-226 by gamma spectroscopy, and for radium-228 if waste soil is generated from PSCs 8, 9, or 17. All samples will be analyzed at an offsite laboratory. Analyses will be performed on these samples to support disposal of the waste material.

Any water collected as a result of decontamination activities will be sampled and analyzed for dissolved and suspended gross alpha/beta activity at an offsite laboratory.

5.2 RADIOLOGICAL SUPPORT - CLEAN AREA

The remaining PSCs (i.e., 3, 4, 5, 8, 9, 15, 16, 25, 29, 32, 40, 41, 42, and 43) are presumed to be free of contamination at this time and are therefore the third priority for radiological support services. To document that these areas are free of residual radioactive materials, protocols have been established following guidelines described in NUREG/CR-5849.

5.2.1 Surface Measurements

- (1) Subdivide the PSC into square grids approximately 10 meters on a side. Clear underbrush to provide a path approximately 1 meter wide along each side of the grid block.
- (2) Survey the path using a gamma scintillation detector, (e.g., 2" x 2" NaI), with ratemeter and audio output, at a foot speed of approximately 2 ft/sec.

- (3) Mark (flag or paint) and note in the field logs each location that indicates an elevated audio signal. The radiological technician should maintain traverse speed, while the support technician marks the elevated reading location.
- (4) At the completion of the grid path survey, return to the marked locations. Using the same detector, but with a scaler, note the level of activity and the extent of activity above background. Record this information using field survey logs.
- (5) If any location is noted that exceeds two times background, designate this area as a "suspect PSC area," and follow protocols defined in Section 5.1.2.
- (6) If all readings are less than two times background, obtain 5-10 soil/sediment samples at randomly selected grid intersection points in the area. Each sample should be approximately 1 kg. The samples will be analyzed for radium-226 by gamma spectroscopy at an offsite laboratory. Radium-228 analyses will also be performed offsite on samples collected from PSCs 8 and 9 due to the abrasive blast grit that may have been disposed at these PSCs.
- (7) After all measurements have been made, perform gamma exposure rate measurements 3 ft above the ground, using a PIC. PIC measurements will be made approximately five per acre, but no less than one per site.

5.2.2 Sediment Sampling

For PSC areas that are or in the outfall from a discharge point, but are not suspected of containing residual radioactive material, verification may be based on analysis of sediment samples taken from these areas. Sediment analysis will be performed in accordance with Section 5.1.5. This is applicable for PSCs 16, 40, and 42. Sediment samples will be collected using a clamshell, mounted to an outrigger on a pontoon boat. An electronic distance meter will be used to identify the sampling locations. All sediment samples will be analyzed for radium-226 by gamma spectroscopy at an offsite laboratory. Five sediment samples will be collected and analyzed from PSCs 16 and 42; 5 sediment samples will be obtained from PSC No. 40.

5.3 BACKGROUND DETERMINATION

To assess the results of the field measurements and the sample analyses, it is necessary to establish the background for the area. This will be accomplished as described below:

5.3.1 In Situ Measurements

- (1) Select approximately 30 locations around NAS Jacksonville that are no closer than 10 meters to an identified PSC (whether radiological or not).

- (2) At each of these locations obtain three readings for each of the following conditions:
- gamma detector and ratemeter with detector approximately 6 inches above ground (scan distance)
 - gamma detector and scaler with detector approximately at ground level
 - beta detector, contact with ground
 - beta detector, facing away from ground
 - alpha detector, contact with ground
 - alpha detector, facing away from ground (don't face sun)

Record all data and determine average for the location.

- Make one PIC measurement at each location.
- (3) Obtain an average, based on a normal distribution (eliminating outliers) for 30 locations. Establish average background for the area. In addition, gamma detector count rates will be correlated with results of soil sample analysis.

5.3.2 Sample Analysis - Soil/Sediment

At the 30 locations, selected in Section 5.3.1, obtain a soil sample approximately 1 kg in size. Submit for offsite analyses for total uranium, radium-226, and radium-228.

5.4 SAMPLE CONTAINER, PRESERVATION, LABELING, AND CHAIN OF CUSTODY

5.4.1 Sample Container and Preservation

Sample volume requirements, preservation techniques, minimum holding times, and container material requirements will be dictated by the media being sampled and the analyses to be performed. Field personnel will be responsible for collecting sufficient volume of each sample in appropriate containers with the proper preservation.

The preparation of all sampling containers and the container types, preservatives, and holding times are specified in the FDER SOPs (DER-QA-001/92), included as Appendix B. Section 4.4 of this FDER SOP contains the recommended container, preservation, and holding times for water, wastewater, soil, and sediment samples. Sample containers will meet all specifications outlined in the above-mentioned procedures.

5.4.2 Sample Custody and Documentation

Sample custody and documentation will be completed in accordance with Section 5.0, "Sample Custody and Documentation," of the FDER SOPs, included in Appendix B.

5.5 DOCUMENTATION OF FIELD ACTIVITIES

Field logbooks will be used for recording all field activities. Entries will include sufficient detail to reconstruct all significant activities. Logbook entries will be completed in accordance with the FDER SOPs, specifically Section 5.0 included in Appendix B. This SOP includes the minimum requirements for recordkeeping.

5.6 SAMPLE TRACKING PROCEDURES

All samples will be tracked from collection to reporting in accordance with the FDER SOPs Section 5.0 contained in Appendix B. The radiological data obtained during this remedial action effort will be validated by ABB-ES.

5.7 SITE-SPECIFIC PROTOCOLS

The protocols to be followed for each of the currently designated PSCs are shown in Table 5-1. Note that for several of the PSCs, more than one protocol will be applicable. Note that, at this time, only PSCs 13, 17, 18, and 26 are planned as radiological suspect areas. All other PSCs will be treated as non-radiological unless the survey of the PSC indicates potential radiological contamination.

The cleanup criteria for NAS Jacksonville will be based on 40 CFR 191 radiological criteria [i.e., 5 picocuries per gram (pCi/g), radium-226, and radium-228 in soil. As low as reasonably achievable principles will be applied during remedial action, including use of the two times background criterion for screening for radioactive contamination and controlling access to all suspect PSC areas.

5.8 DELIVERABLES

A report to ABB-ES and the Navy will be submitted after completion of the work. The reports will include the field log sheets (encompassing field forms and drawings) completed during the remedial action. Specifically, the log sheets will indicate the location of elevated readings of radionuclides from the gamma walkover survey, the elevated gamma walkover survey reading, whether excavation was performed, sampling locations and the associated sample numbers, and the gamma survey reading and location after remediation. The laboratory will provide data sheets with the associated sample number. The log sheets will also provide additional information in accordance with the FDER SOPs.

6.0 WASTE MANAGEMENT

Waste management will be performed as directed by the Navy Public Works Division. Waste management practices, as defined in the Program Hazardous Waste Management Plan, will be used as guidance and appropriately followed for this work.

All radioactive materials exhumed from the PSCs, and all potentially contaminated materials generated during site support activities will be containerized. All packaged waste will be labeled with the container's contents, date generated, and location from where it was derived. To

Table 5-1
Site-Specific Radiological Survey Protocols

<u>PSC NO.</u>	<u>SITE IDENTIFICATION</u>	<u>APPLICABLE SECTION</u>
3.	Wastewater Treatment Plant (WWTP); Ex-sludge Disposal Area	5.2.1
4.	Pine Tree Planting Area	5.2.1
5.	Shoreline Fill West of Fuel Barge Deck	5.2.1
8.	Vacant Lot, East of Gas Hill	5.2.1
9.	Old Disposal Area, East of Fuel Farm	5.2.1
13.	Radium Paint Disposal Pit	5.1.1, 5.1.2, 5.1.3
15.	Solvent and Paint Sludge Disposal Area	5.2.1
16.	Storm Sewer Discharge, Black Point	5.2.2
17.	Glass Bead Disposal Area, Above Waterline	5.1.1, 5.1.2, 5.1.3
	Glass Bead Disposal Area, Below Waterline	5.1.4
18.	Radioactive Waste Disposal Area	5.1.1, 5.1.2, 5.1.3
25.	Bldg. 2038 NH - Ex-radwaste Storage Area	5.2.1
26.	Old Main Registered Disposal Area	5.1.1, 5.1.2, 5.1.3
	Moat Around Disposal Area	5.1.4
29.	Organic Disposal Area	5.2.1
32.	Ex-Base Landfill	5.2.1
40.	Ex-East IWWTP Discharge Area	5.2.2
41.	Domestic Waste Sludge Drying Beds	5.2.1
42.	Polishing Pond	5.2.2
43.	Industrial Waste Sludge Drying Beds	5.2.1

minimize the amounts of materials that must eventually be disposed, waste minimization practices will be implemented during operations. These practices will include, but not be limited to:

- (1) No extraneous materials taken into contamination control areas;
- (2) Decontamination and free release of equipment used to support onsite activities, to the extent practicable;
- (3) Use of consumables that can be compacted or otherwise volume reduced, to the extent practicable

All radioactive waste materials will be packaged in 17H (55-gallon) drums. Assessment for exhumed materials will be based on analytical laboratory analysis of samples, obtained as described in Section 5.1.2. The drummed waste material will be temporarily stored inside the existing fenced area at PSC 26, per the Navy's direction. For the miscellaneous wastes generated to support onsite activities, radiological assessment will be based upon a combination of external radiological measurements on the waste package, process knowledge, and dose-activity conversion factors for the package. Information on drum contents in the form of laboratory data sheets and field log sheets (encompassing field forms) will be provided to the Navy Public Works Division. All radiological wastes will be disposed as directed by the Navy Public Works Division.

If the analytical results from the laboratories show the drummed waste meets the definition of a mixed waste, then the waste will be managed as a Resource Conservation and Recovery Act (RCRA) waste, and requirements of 40 CFR 262 will be followed. The programmatic Hazardous Waste Management Plan, Sections 3.4.1 and 4.0, outlines the appropriate waste management procedures that will be followed.

7.0 SAFETY AND HEALTH

A Program Safety and Health Plan (PSHP) defines policies for work on the NAVYRAC Project. A Site Safety and Health Plan (SSHP) has been prepared for the NAS Jacksonville site. A Task-Specific Safety and Health Plan, Addendum No. 1 to the SSHP, defines task-specific requirements for the radiological remediation for NAS Jacksonville.

8.0 QUALITY CONTROL PLAN

QC samples will be collected during radiological sampling activities and will be used as a means of evaluating data quality in terms of precision and accuracy. QC checks also verify that sampling, handling, and analysis does not introduce contaminants into the sampling process. Table 8-1 is included to show the minimum field QC sampling frequency. A site specific QC Plan that addresses the radiological remediation is attached to this work plan as Appendix C.

Table 8-1 Minimum Field QA/QC Sample Frequency

Sample Type	Minimum Frequency	Source
Trip blank	1 per shipment or as directed by the Project Manager (PM) (for VOC analysis only)	Laboratory provides sealed and preserved
Equipment wash blank	1 per week or per sampling event	Collect final rinsate from equipment decontamination
Field blank	To be determined by PM	Collect organic-free/DI water sample in the field
Field duplicates	5% or 1/20 per matrix per day	Collect duplicate samples
Split	As directed by PM	Laboratory splits sample and ships to independent laboratory

APPENDIX A

TECHNICAL SPECIFICATION FOR CIVIL SURVEYING


DEPARTMENT OF THE NAVY

SOUTHERN DIVISION

TECHNICAL SPECIFICATION

FOR

SURVEYING SERVICES

0	6/16/94	ISSUED FOR CONSTRUCTION	MMB	YMS	AK	PLB
NO.	DATE	REVISION	BY	CHK'D	SUPV	PE
ORIGIN		SURVEYING SERVICES	JOB NO. 22567			
			TECHNICAL SPECIFICATION		REV.	
			201-SP006-001		0	
			SHEET 1 OF 19			

TECHNICAL SPECIFICATION

FOR

SURVEYING SERVICES

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TECHNICAL SPECIFICATION
FOR
SURVEYING SERVICES

1.0 GENERAL

Not all activities defined herein may be required. Only those activities required in the applicable Subcontract Scope of Work and design drawings for specific service shall apply.

2.0 ABBREVIATIONS

The abbreviations listed below, where used in this specification, shall have the following meaning:

NOAA	National Oceanic and Atmospheric Administration
USGS	United States Geological Survey
ASCII	American Standard Code for Information Interchange
CADD	Computer Aided Design and Drafting
DOS	Disk Operating System
IGDS	Interactive Graphic Design Software
NAD	North American Datum

3.0 QUALITY STANDARDS

3.1 Unless otherwise specified or shown, the following code and standard of the latest issue at the time of bid shall apply to the extent indicated herein:

NOAA	Classification, Standards of Accuracy, and General Specifications of Geodetic Control Surveys, 1974
------	---

- 3.2 Standards of accuracy for all survey work shall be in accordance with NOAA standards and the minimum accuracy set forth below. The horizontal accuracy for location of all grid intersections and planimetric features shall be (\pm) 0.1 feet. The horizontal accuracy for boundary surveys shall be one in ten thousand (1:10,000). The vertical accuracy for ground surface elevations shall be (\pm) 0.1 feet. Bench mark elevation accuracy and elevation accuracy of other permanent items (e.g., structures, pavements, etc.) shall be (\pm) 0.01 feet.
- 3.3 All work shall be performed under the direct supervision of a Land Surveyor registered in the state where the work is located. Survey crew personnel shall be competent and experienced in performing land survey work.
- 3.4 Horizontal ground control surveys shall be based on NAD 83. State Plane coordinates shall be computed for NAD 83, NAD 27, and any applicable Bechtel project coordinate system. Vertical ground control surveys shall be based on NAD 29. Elevations shall be computed for NAD 29. All coordinates and elevations shall be stated in English units of measure (Decimal Feet).
- 3.5 Drawings and calculations (except for originals) submitted by the Subcontractor shall be signed, sealed, and certified by a Land Surveyor registered in the state where the work is located.

4.0 SUBMITTALS

4.1 Engineering documentation requirements are summarized on the Subcontractor Submittal Requirements Summary, Exhibit "F", and are augmented by detailed requirements listed herein. Bechtel will determine if documentation is complete as submitted by the Subcontractor, and reserves the right to reject and require resubmittal of any submittal that does not meet the Subcontract requirements.

4.2 Submittal of data on magnetic storage media shall be accompanied by a hardcopy list of the media contents and a letter of transmittal including the following:

- Subcontract number
 - A description of contents per the Subcontract
 - Number and type of items (floppies, etc.)
 - Note if the submittal is a resubmittal

5.0 MATERIALS

5.1 CONCRETE MONUMENTS

Concrete monuments shall be as shown on Attachment A or an equivalent submitted to Bechtel for review. Concrete shall have a minimum 28-day compressive strength of 3000 psi.

5.2 MAGNETIC MEDIA

Floppy Disk:

5-1/4" (1.2 MB) or 3-1/2" (1.44 MB), DOS 3.3 or higher.
DOS backup for files greater than 1.44 MB.

5.3 FIELD BOOKS AND DATA SHEETS

The necessary field data shall be recorded by the surveyor in a standard field book or on standard surveying data sheets using generally accepted surveying field note recording practices. The data shall be clear, neat, and easily reproducible. Bechtel may accept highly legible copies of field data with the written consent of the Bechtel Site Superintendent.

5.4 IRON PINS

Iron pins shall consist of an 18-inch length of reinforcement steel or equivalent with a minimum diameter of 5/8 inch.

5.5 METAL MONUMENTS

Metal monuments shall be as indicated on the design drawings.

5.6 WOODEN HUBS

Wooden hubs shall be 2 inch x 2 inch, at least 8 inches long, milled from solid lumber, and shall be pointed on one end. (Approved substitutes may be used with the permission of the Bechtel Site Superintendent.)

5.7 WOODEN STAKES

Wooden stakes shall be 1 inch x 2 inches, at least 3 feet long, milled from solid lumber, and shall be pointed on one end. Wooden stakes shall be clearly marked with bright orange weatherproof flagging and paint. (Approved substitutes may be used with the permission of the Bechtel Site Superintendent.)

5.8 MISCELLANEOUS

Miscellaneous materials (e.g., P-K nails, flagging, etc.) shall be of the type and quality normally used for land survey work.

6.0 EQUIPMENT

- 6.1 The Subcontractor shall maintain sufficient equipment, materials, parts, tools, and supplies to meet the requirements of the work. Surveying equipment shall be subject to inspection by Bechtel and, if deemed unsatisfactory, shall be removed from the site and replaced by satisfactory equipment. Surveying instruments, (level, transit, EDM, etc.) shall have been inspected and calibrated by an authorized manufacturer's representative not more than six months prior to the survey; the Subcontractor shall submit a certificate of compliance for each instrument to Bechtel at the beginning of the Subcontract.

7.0 FIELD OPERATIONS

- 7.1 The Subcontractor shall verbally notify Bechtel in advance of commencing survey work and shall provide a minimum of 24 hours notice to Bechtel when it is necessary to enter private property.
- 7.2 When any survey work is conducted during the construction period, the Subcontractor shall employ all possible means to minimize interference with construction work by others. Any damage to facilities caused by the Subcontractor shall be repaired or replaced at the expense of the Subcontractor.

8.0 OFFICE WORK

- 8.1 For digital maps, design drawing, data, and other surveying, the subcontractor may be required to provide and accept design files for Intergraph and/or Auto CADD.
- 8.2 Digital map data shall be input into a SINGLE 3D design file and shall be placed on designated levels in accordance with a Bechtel CADD Level Index Record as described in Exhibit A.
- 8.3 The digital map shall utilize state plane coordinates based on NAD 27 and elevations based on NAD 29.
- 8.4 Map data shall be input into the design file at a scale ratio of 1:1 and any patterning, cells, text, or other scale dependent items shall be sized for a plot scale of 1"= 20' and a minimum lettering size of 1/8" in height.

- 8.5 Line Strings shall be used for linear features; shapes for enclosed features (buildings, ponds, etc...); arcs should only be used when standard radii are given (curb intersections, etc...). Curve Strings shall not be used.
- 8.6 Linear patterning of features such as railroads, fences, etc... is not required. If any linear patterns are used they must retain the original element (class 5) intact when pattern display is turned off. All symbols shall be cells from the Bechtel-provided cell library.
- 8.7 Active attributes (color, line code, weight, font, etc...) shall be set to zero unless otherwise specified in Exhibit A. Map features shall not be clipped around text or symbols.
- 8.8 All design files shall be based on the state plane coordinate system using the global origin and working units setup in the Bechtel-provided "state" STP design file.
- 8.9 The Subcontractor shall place his own drawing format around the digital map with a legend, date, notes on accuracy, and any miscellaneous notes.
- 8.10 The Subcontractor shall submit one digital copy of the topographic map to Bechtel for review and comment. Once Subcontractor has incorporated comments, he shall submit one digital copy of the topographic map to Bechtel along with a Letter of Certification for the map that is signed, sealed, and certified by a registered Land Surveyor.

- 8.11 The Subcontractor shall reduce field notes and perform all calculations required to develop the information needed in each type of survey. Field books and data sheets shall become the property of Bechtel at the close of the Subcontract unless the Bechtel Site Superintendent agrees to suitable substitutes.
- 8.12 When electronic equipment is used to store survey data, both a printout of the data (with notations to identify and explain data) and magnetic media containing the data in ASCII format shall be submitted.

9.0 BOUNDARY SURVEYS

9.1 FIELD OPERATIONS

- 9.1.1 The Subcontractor shall perform surveys and deed research necessary to define the property boundaries for the properties shown on the drawings and as directed by Bechtel.
- 9.1.2 Coordinates for all boundary surveys shall be based on the local state plane coordinate system.
- 9.1.3 Concrete or metal monuments or iron pins, as indicated on the drawings, shall be set at each property corner not having a permanent marker.
- 9.1.4 Pertinent information obtained and/or established during the boundary surveys shall be shown on the drawings and submitted to Bechtel on magnetic media for review in accordance with this specification.

9.2 OFFICE WORK

9.2.1 Drawings for boundary surveys shall show:

- Major structures (buildings, storage tanks, etc.)
- Property corners and lines
- Tie to closest street intersection
- State plane coordinates for two property corners
- Property line dimensions, political boundaries, bearings and other miscellaneous data pertinent to the boundary survey

10.0 GRID SURVEYS

10.1 FIELD OPERATIONS

- 10.1.1 The Subcontractor shall perform grid surveys within the limits and at the intervals shown on the drawings or as directed by Bechtel.
- 10.1.2 Unless noted otherwise, the intersection of the grid lines shall be marked with wooden hubs driven flush with the ground to a minimum depth of 8 inches. Where wooden hubs can not be driven, P.K. nails or chiseled crosses shall be used to establish grid intersection points. The coordinates and elevations of the hubs at the grid points shall be established, recorded, and marked on wooden stakes driven within 12 inches of said hubs. The Subcontractor shall establish a bench mark within the property at the location

shown on the drawings or as directed by Bechtel. Spray paint marking on buildings, structures, or pavements shall not be used.

- 10.1.3 Where the grid intersection location is obstructed by physical barriers, wooden hubs shall be set on the grid line to mark the obstruction. The coordinates and ground surface elevation at these points shall be established, recorded, and marked as described above.
- 10.1.4 Coordinates for grid surveys shall be based on the coordinate system shown on the drawings; at least two coordinates for grid surveys shall be tied into the local state plane coordinate system.
- 10.1.5 The grid shall be referenced to permanent features within or immediately adjacent to the survey area so that the grid may be readily reestablished in the event that it is removed or disturbed.
- 10.1.6 Pertinent data and information obtained and/or established during the grid surveys shall be shown on the drawings and submitted to Bechtel on magnetic media for review in accordance with this specification.

10.2 OFFICE WORK

- 10.2.1 Drawings and documentation for grid surveys shall show:

- Plan of area grid
- Coordinates and elevations of grid intersection points and of other points along the grid lines shown in tabular form
- Grid lines and all other miscellaneous data pertinent to the grid survey
- Property lines and major structures (buildings, storage tanks, etc.)
- Ties to the state plane coordinate system sufficient to enable the survey to be reestablished at a future date

11.0 CONTOUR SURVEYS

11.1 FIELD OPERATIONS

- 11.1.1 The Subcontractor shall perform contour surveys within the limits shown on the drawings and as directed by Bechtel
- 11.1.2 Sufficient surface elevations shall be measured to define the contour interval required on the drawings and to define all breaks in the terrain.
- 11.1.3 Pertinent data and information obtained and/or established during the contour surveys shall be shown on the drawings and submitted to Bechtel on magnetic media for review in accordance with this specification.

11.2 OFFICE WORK

- 11.2.1 Drawings for contour surveys shall show:

- Property lines and major structures (buildings, storage tanks, etc.)
- Contours of the terrain and elevations of breaks in the terrain
- Bench marks and all other miscellaneous data pertinent to the contour survey
- Ties to the state plane coordinate system sufficient to enable the survey to be reestablished at a future date

12.0 PLANIMETRIC SURVEYS

12.1 FIELD OPERATIONS

- 12.1.1 The Subcontractor shall perform planimetric surveys within the limits shown on the drawings and as directed by Bechtel.
- 12.1.2 All planimetric features shall be located, including but not limited to paved surfaces, vegetation, fences, power poles, walkways, underground utilities, structures and all other obstructions.
- 12.1.3 Pertinent data and information obtained and/or established during the planimetric surveys shall be shown on the drawings and submitted to Bechtel on magnetic media for review in accordance with this specification.

12.2 OFFICE WORK

- 12.2.1 Drawings for planimetric surveys shall show:

- Property lines and major structures (buildings, storage tanks, etc.)
- All planimetric features, including but not limited to, paved surfaces, vegetation, fences, power poles, walkways, underground utilities, structures and all other obstructions
- Bench marks and all miscellaneous data pertinent to the planimetric survey
- Ties to the state plane coordinate system sufficient to enable the survey to be reestablished at a future date

12.2.2 An inventory of all trees and plants, shown on the planimetric survey, shall be recorded by a nurseryman and will include the correct botanical names, common names, sizes, an estimation of the replacement size and cost, condition and identification number. The replacement size shall be the largest size, not to exceed the existing plant size, that can be planted without the use of heavy equipment. The planting inventory shall be as shown in Attachment B. The inventory shall be completed and submitted to Bechtel unless provided to Bechtel on the planimetric drawings.

13.0 CONSTRUCTION SURVEYS

13.1 The Subcontractor shall perform construction surveys and related calculations as directed by Bechtel.

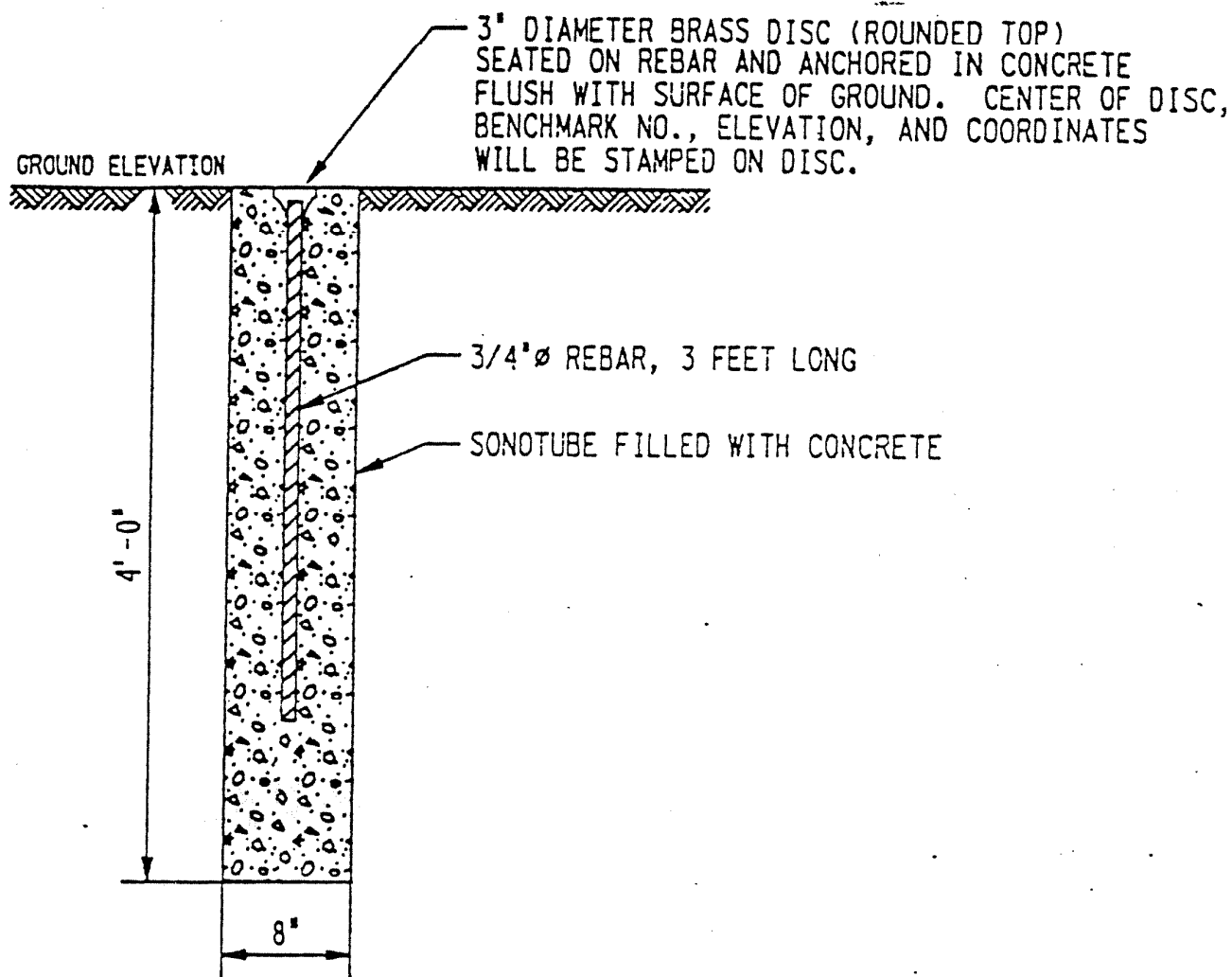
13.2 Pertinent data and information obtained and/or established during the construction surveys shall be submitted as directed by the Bechtel Site Superintendent.

13.3 Drawings for construction surveys, if required by the Bechtel site superintendent, shall be submitted to Bechtel on magnetic media for review in accordance with the requirements of this specification.

14.0 ADDITIONAL DATA FILES

Any digital models used for contour generation shall be provided to Bechtel, including any of the following:

<u>FILE</u>	<u>DESCRIPTION</u>
.DAT	ASCII point data (east, north, elevation).
.XYZ	Intergraph binary point data.
.TIN	Binary triangle data.
.TTN	Binary terrain model data.
.DTM	Binary terrain model data.



CONCRETE SURVEYING MONUMENT DETAIL

Attachment A

Specification 201-SP006-001

ATTACHMENT B

QUANTITY	I.D. NO.	BOTANICAL NAME	COMMON NAME	EXISTING SIZE	REPLACEMENT SIZE	REPLACEMENT COST	REMARKS & CONDITION
2	S1	Taxus Densa	Spreading Yew	6'	5'	\$ 169.00	Good
1	S2	Syringa Vulgaris	Lilac	10'	8'	205.00	Good
1	S3	Syringa Vulgaris	Lilac	12'	6'	140.00	Fair
1	S4	Paulownia Tomentosa	Paulownia	10'	0	-0-	Poor
1	S5	Rosa Species	Rose	6'	4'	30.00	Fair
3	T1	Tsuga Canadensis	Canadian Hemlock	12'	8-10'	219.00	Fair
2	T2	Acer Planatoides	Norway Maple	12'	6'	1000.00	Good
3	T3	Pseudotsuga Taxifolia	Douglas Fir	6'	4'	99.00	Fair
1	T4	Pseudotsuga Taxifolia	Douglas Fir	'15	12'	388.00	Good
EXAMPLE ASSOCIATES Engineers & Surveyors 1000 Union Avenue Middlesex, New Jersey 08846 (201) 666-7777			Owner: Joseph N. and Susan Hughes				
STEPHEN EXAMPLE, L.S., Lic. 5000 CLYDE EXAMPLE, P.E., L.S., Lic. 200			PLANT INVENTORY 1999 Wilkinson Road Maywood, New Jersey				



**NAVY RAC CADD LEVEL INDEX
BECHTEL ENVIRONMENTAL, INC.
OAK RIDGE, TN**

LEVEL	DESCRIPTION	LEVEL	DESCRIPTION
1	*	33	SPOT ELEVATIONS
2	*	34	*
3	* well locations	35	*
4		36	*
5	*	37	*
6	*	38	RAILROADS
7	* contamination areas	39	PRIMARY ROADS
8	*	40	secondary roads
9	*	41	PRIMARY STRUCTURES
10	DRAWING BORDER & TITLE BLOCK INFO., NORTH ARROW, SCALE	42	secondary structures
11	*	43	FENCES
12	*	44	PRIMARY ABOVE GROUND UTILITIES
13	*	45	secondary above ground utilities
14	*	46	*
15	REVISION CLOUDS AND TRIANGLES	47	PRIMARY BELOW GROUND UTILITIES
16	MONUMENTS AND BENCHMARKS	48	secondary below ground utilities
17	PRIMARY GRID LINES	49	*
18	PRIMARY GRID LINES	50	PRIMARY LANDSCAPING
19	secondary grid lines	51	*
20	secondary grid labels	52	*
21	*	53	*
22	*	54	PRIMARY TEXT
23	*	55	secondary text & dimensioning
24	*	56	*
25	MAJOR PROJECT SPECIFIC BOUNDARIES	57	*
26	minor project specific boundaries	58	*
27	LOCAL BOUNDARIES (PROPERTY LINES, ETC.)	59	*
28	PRIMARY HYDROLOGY	60	*
29	secondary hydrology	61	*
30	INDEX CONTOURS & TEXT	62	data to be saved, but not viewed or plotted
31	PRIMARY CONTOURS	63	EMPTY
32	secondary contours	64	* = PROJECT SPECIFIC

PART VI (or EXHIBIT "F")
SUBCONTRACTOR SUBMITTAL REQUIREMENTS SUMMARY

EDPI-4.58-01

Exhibit A

Page 1 of 2

SUBMITTAL SCHEDULE

Prior to Fabrication
 Prior to Shipment
 Prior to Balance of Payment
 Per S/C Schedule
 Prior to Mobilization
 Prior to Commencing Work
 Prior to Use
 Prior to Purchase
 Prior to Progress Payment
 for Each Specific Task
 As Required
 Calendar Days After Notice of Award

SUBMITTAL TYPE REQUIRED

O Original
 P Prints/Photocopies
 T Transparencies
 M Microfilm
 PH Photographs
 FD Floppy Disk
 S Sample
 (2) Number of Copies

DISTRIBUTION DESIGNATION

SC Submittal Coordinator
 CA Contract Administrator
 SS Site Superintendent
 ET Engineering & Technology
 WM Waste Management
 CO Construction
 SH Safety & Health
 PR Procurement
 EC Environmental Compliance
 DM Data Management
 QA Quality Assurance
 AA

BECHTEL TRACKING CODES

CCR Communications Control Register
 SDR Supplier Document Register

NOTICES


1. To each item submitted, attach a copy of this form and circle the title of the item being submitted.
2. Failure to submit required submittals as delineated on this form may result in withholding of payment in accordance with provisions of the subcontract.

SUBMIT TO:

Joe Johnson
 Contract Administrator (CA)
 Field Office Trailer
 Anywhere NJ 01234

OR

Mary Jackson
 Submittal Coordinator (SC) Job 22222 QUIKRAP
 Bechtel National Inc.
 151 Lafayette Dr.
 Oak Ridge TN 37831-0350

Item No./ Submittal Title	Clause, Specification, or Scope of Work Paragraph	Submit to	Submittal Codes		FOR BECHTEL USE ONLY		
			Schedule	(No.) and Type	Send to/for		Tracking Codes
					Review	Info	
Construction							
1.1 Authorized Representative	Part II, Clause 9	SC	M	O		CO	CCR
1.2 Notice of Labor Dispute (actual or potential)	Part I, Clause 30	SC	Z	O	SS	CO	CCR
1.3 Payrolls and Basic Records	Part I, Clause 35	CA	Y	O	SS	SC	CCR
1.4 Statement and Acknowledgement Form 1413 (Lower tier Subcontractors)	Part I, Clause 38	SC	Z	O		CO	CCR
1.5 Release and Certification of Final Payment	Part II, Clause 37	CA	B	O	SS	SC	SDR
2. Safety & Health							
2.1 Safety Representative Letter	Part II, Cl.		S	O		SH	CCR
2.2 Accident and Injury Report			A	O		SH	CCR
			A	(2)P		SS	CCR
2.3 Material Safety Data Sheets		SC	Z	O	SS		CCR
2.4 Valid First Aid/CPR Card	Part II, Clause 59	SC	W	P	SH		SDR
2.5 List of Subcontractor Personnel to Work Onsite and their addresses	Part II, Clause 59	SC	W	(4)P		SH	SDR
2.6 Proof of OSHA Training	Part II, Clause 59	SC	W	P	SH	SS	SDR
			W	P			SDR
	Project Number	Project Name	Subcontract Number	SSRS Rev. No.	Date:		
	XXXXX	XXXXXX	XXX-SC-XXX		Page 1 of 2		

**PART VI (or EXHIBIT "F")
SUBCONTRACTOR SUBMITTAL REQUIREMENTS SUMMARY**

EDPI-4.58-01

Exhibit A

Page 2 of 2

SUBMITTAL SCHEDULE

Prior to Fabrication
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SC Submittal Coordinator
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BECHTEL TRACKING CODES

CCR Communications Control Register
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Item No./ Submittal Title	Clause, Specification, or Scope of Work Paragraph	Sub- contractor send Submittal to	Submittal Codes		FOR BECHTEL USE ONLY		
			Schedule	(No.) and Type	Send to/for		Tracking Codes
					Review	Info	
2.7 Physician's Statement	Part II, Clause 59	SC	W	O	SH		SDR
2.8 Letter Confirming Intent to Adopt <i>(insert project's name)</i> Safety and Health Program	Part II, Clause 59	SC	W	O		SH	CCR
3. Waste Management							
3.1 Spill Contingency	Part II, Clause 62	SC	S,W	P	WM		SDR
3.2 Hazardous Waste Transport Permits	Part II, Clause 62	SC	S,W	P	WM		SDR
3.3 Hazardous Waste Manifests	Part II, Clause 62	SC	W	P	WM		SDR
3.4 Driver Training	Part II, Clause 62	SC	S,W	P	WM		SDR
3.5 HAZMAT Employee Certification	Part II, Clause 62	SC	W	P	WM		SDR
			W	P		SH	SDR
3.6 Vehicle Inspection Reports	Part II, Clause 62	SC	W	P	WM	SH	SDR
			W	P		SH	SDR
4. Procurement							
4.1 Subcontracting Plan	Part I, Clause 25		Z	O	PR		CCR
			Z	P		SS	CCR
4.2 Insurance Certificate			W	O	PR		CCR
			W	P		SS	CCR
4.3 Performance and Payment Bonds			W	O	PR		CCR
5. Engineering & Technology							
5.1 Bentonite	Spec. 31-5.2.1	SC	M	Q,S	ET	SS	SDR
5.2 MSDS for Concrete Patch	Spec. 31-5.3.1 Spec. 32-5.4.1	SC	M	P	ET	SS	SDR
5.3 Detail of Well Cap	Spec. 33.5.8	SC	M	Q	ET	SS	SDR
5.4 Field Permeability Data	Scope 1.3.2	SC	Z	O	ET	SS	SDR
	Project Number XXXXXX	Project Name XXXXXX	Subcontract Number XXX-SC-XXX	SSRS Rev. No.	Date:		
Page 2 of 2							


PART VI (or EXHIBIT "F")
SUBCONTRACTOR SUBMITTAL REQUIREMENTS SUMMARY

SUBMITTAL SCHEDULE		SUBMITTAL TYPE REQUIRED	DISTRIBUTION DESIGNATION	BECHTEL TRACKING CODES
F	Prior to Fabrication	O Original	SC Submittal Coordinator	CCR Communications Control Register
	Prior to Shipment	P Prints/Photocopies	CA Contract Administrator	SDR Supplier Document Register
	Prior to Balance of Payment	T Transparencies	SS Site Superintendent	
	Per S/C Schedule	M Microfilm	ET Engineering & Technology	
M	Prior to Mobilization	PH Photographs	WM Waste Management	
W	Prior to Commencing Work	FD Floppy Disk	CO Construction	
U	Prior to Use	S Sample	SH Safety & Health	
X	Prior to Purchase	Q _____	PR Procurement	
Y	Prior to Progress Payment	(2) Number of Copies	EC Environmental Compliance	
	for Each Specific Task		DM Data Management	
Z	As Required		QA Quality Assurance	
5	Calendar Days After Notice of Award		AA _____	

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2. Failure to submit required submittals as delineated on this form may result in withholding of payment in accordance with provisions of the subcontract.

SUBMIT TO: Joe Johnson Contract Administrator (CA) Field Office Trailer Anywhere NJ 01234	OR	Mary Jackson Submittal Coordinator (SC) Job 22222 QUIKRAP Bechtel National Inc. 151 Lafayette Dr. Oak Ridge TN 37831-0350
--	----	---

Item No./ Submittal Title	Clause, Specification, or Scope of Work Paragraph	Submit to	Submittal Codes		FOR BECHTEL USE ONLY			
					Send to/for		Tracking Codes	
			Schedule	(No.) and Type	Review	Info		
1. Construction								
1.1 Authorized Representative	Part II, Clause 9	SC	M	O		CO	CCR	
1.2 Notice of Labor Dispute (actual or potential)	Part I, Clause 30	SC	Z	O	SS	CO	CCR	
1.3 Payrolls and Basic Records	Part I, Clause 35	CA	Y	O	SS	SC	CCR	
1.4 Statement and Acknowledgement Form 1413 (Lower tier Subcontractors)	Part I, Clause 38	SC	Z	O		CO	CCR	
1.5 Release and Certification of Final Payment	Part II, Clause 37	CA	B	O	SS	SC	SDR	
2. Safety & Health								
2.1 Safety Representative Letter	Part II, Clause 59	SC	5	O		SH	CCR	
2.2 Accident and Injury Report	Part II, Clause 59	SC	A A	O (2)P		SH SS	CCR CCR	
2.3 Material Safety Data Sheets	Part I, Clause 51	SC	Z	O	SS		CCR	
2.4 Valid First Aid/CPR Card	Part II, Clause 59	SC	W	P	SH		SDR	
2.5 List of Subcontractor Personnel to Work Onsite and their addresses	Part II, Clause 59	SC	W	(4)P		SH	SDR	
2.6 Proof of OSHA Training	Part II, Clause 59	SC	W W	P P	SH	SS	SDR SDR	
	Project Number	Project Name	Subcontract Number	SSRS Rev. No.		Date:		
	22567	NAVYRAC	201-SC-006	0		06/27/94		
							Page 1 of 3	

PART VI (or EXHIBIT "F") **SUBCONTRACTOR SUBMITTAL REQUIREMENTS SUMMARY**

SUBMITTAL SCHEDULE

F Prior to Fabrication
 Prior to Shipment
 Prior to Balance of Payment
 A Per S/C Schedule
 M Prior to Mobilization
 W Prior to Commencing Work
 U Prior to Use
 X Prior to Purchase
 Y Prior to Progress Payment
 for Each Specific Task
 Z As Required
 5 Calendar Days After Notice of Award

SUBMITTAL TYPE REQUIRED


O Original
 P Prints/Photocopies
 T Transparencies
 M Microfilm
 PH Photographs
 FD Floppy Disk
 S Sample
 Q _____
 (2) Number of Copies

DISTRIBUTION DESIGNATION

SC Submittal Coordinator
 CA Contract Administrator
 SS Site Superintendent
 ET Engineering & Technology
 WM Waste Management
 CO Construction
 SH Safety & Health
 PR Procurement
 EC Environmental Compliance
 DM Data Management
 QA Quality Assurance
 AA _____

BECHTEL TRACKING CODES

CCR Communications Control Register
 SDR Supplier Document Register

Item No./ Submittal Title	Clause, Specification, or Scope of Work Paragraph	Sub- contractor send Submittal to	Submittal Codes		FOR BECHTEL USE ONLY		
			Schedule	(No.) and Type	Send to/for		Tracking Codes
					Review	Info	
2.7 Physician's Statement	Part II, Clause 59	SC	W	O	SH		SDR
2.8 Letter Confirming Intent to Adopt <u>(insert project's name)</u> Safety and Health Program	Part II, Clause 59	SC	W	O		SH	CCR
3. Waste Management							
3.1 Spill Contingency	Part II, Clause 62	SC	S,W	P	WM		SDR
3.2 Hazardous Waste Transport Permits	Part II, Clause 62	SC	S,W	P	WM		SDR
3.3 Hazardous Waste Manifests	Part II, Clause 62	SC	W	P	WM		SDR
3.4 Driver Training	Part II, Clause 62	SC	S,W	P	WM		SDR
3.5 HAZMAT Employee Certification	Part II, Clause 62	SC	W W	P P	WM	SH	SDR SDR
3.6 Vehicle Inspection Reports	Part II, Clause 62	SC	W W	P P	WM	SH SH	SDR SDR
4. Procurement							
3 4.1 Subcontracting Plan	Part I, Clause 25	SC	Z Z	O P	PR	SS	CCR CCR
4.2 Insurance Certificate	Part II, Clause 20	SC	W W	O P	PR	SS	CCR CCR
4.3 Performance and Payment Bonds	Part II, Clause 30	SC	W	O	PR		CCR
4 Engineering							
5.1 Certificate of Compliance for Surveying Instruments	Tech. Spec., 6.1	SA	Z	U	FE	CO	SDR
5.2 Preliminary Survey Information when requested by Bechtel	Tech. Spec., 8.12	SA	Z	FD/Q	FE	CO	SDR
5.3 Field Books and Data Sheets or Suitable Substitutes	Tech. Spec., 8.11	SA	Z	O/P	FE	CO	SDR
	Project Number	Project Name	Subcontract Number	SSRS Rev. No.	Date:		
	22567	NAVYRAC	201-SC-006	0	06/27/94		
Page 2 of 3							

PART VI (or EXHIBIT "F")
SUBCONTRACTOR SUBMITTAL REQUIREMENTS SUMMARY

SUBMITTAL SCHEDULE

F Prior to Fabrication
 Prior to Shipment
 Prior to Balance of Payment
 A Per S/C Schedule
 M Prior to Mobilization
 W Prior to Commencing Work
 U Prior to Use
 X Prior to Purchase
 Y Prior to Progress Payment
 for Each Specific Task
 Z As Required
 5 Calendar Days After Notice of Award

SUBMITTAL TYPE REQUIRED

O Original
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 S Sample
 Q _____
 (2) Number of Copies


DISTRIBUTION DESIGNATION

SC Submittal Coordinator
~~CA~~ Contract Administrator
 SS Site Superintendent
 ET Engineering & Technology
 WM Waste Management
 CO Construction
 SH Safety & Health
 PR Procurement
 EC Environmental Compliance
 DM Data Management
 QA Quality Assurance
~~EA~~ ~~E~~

BECHTEL TRACKING CODES

CCR Communications Control Register
 SDR Supplier Document Register

FE FIELD ENGINEERING

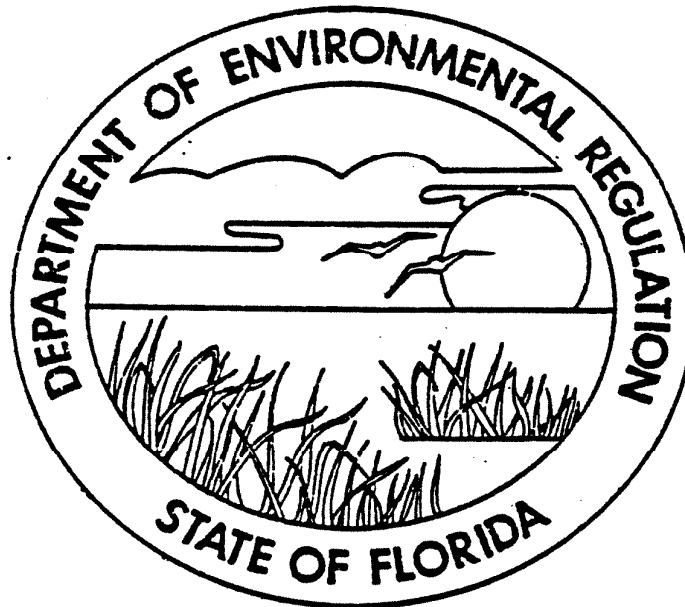
Item No./ Submittal Title	Clause, Specification, or Scope of Work Paragraph	Sub- contractor send Submittal to	Submittal Codes		FOR BECHTEL USE ONLY		
			Schedule	(No.) and Type	Send to/for		Tracking Codes
					Review	Info	
Δ 5.4 Boundary Survey Dwgs	Tech. Spec., 9.0	SA	Z	FD/Q	FE	CO	SDR
Δ 5.5 Grid Survey Dwgs	Tech. Spec., 10.0	SA	Z	FD/Q	FE	CO	SDR
Δ 5.6 Contour Survey Dwgs	Tech. Spec., 11.0	SA	Z	FD/Q	FE	CO	SDR
Δ 5.7 Planimetric Survey Dwgs	Tech. Spec., 12.0	SA	Z	FD/Q	FE	CO	SDR
Δ 5.8 Inventory of Trees and Plants (if not included on planimetric)	Tech. Spec., 12.2	SA	Z	FD/Q	FE	CO	SDR
Δ 5.9 Construction Surveys	Tech. Spec., 13.0	SA	Z	FD/Q	FE	CO	SDR
<div style="display: flex; justify-content: space-between; align-items: center;">  <div> <p>Project Number 22567</p> <p>Project Name NAVYRAC</p> <p>Subcontract Number 201-SC-006</p> <p>SSRS Rev. No. 0</p> <p>Date: 06/27/94</p> <p>Page 3 of 3</p> </div> </div>							

APPENDIX B

**FLORIDA DEPARTMENT OF ENVIRONMENTAL REGULATION
STANDARD OPERATING PROCEDURES**

DEPARTMENT OF ENVIRONMENTAL REGULATION
STANDARD OPERATING PROCEDURES
FOR
LABORATORY OPERATIONS AND SAMPLE COLLECTION ACTIVITIES

DER - QA-001/92



Florida Department of Environmental Regulation
2600 Blair Stone Road
Tallahassee, Florida 32399-2400

QUALITY ASSURANCE SECTION

September 30, 1992

NOTICE OF CHANGE IN SOP FOR GROUNDWATER FILTRATION

As of April 14, 1994, Chapter 17-520, F.A.C. in a Guidance Document titled "Determining Representative Ground Water Samples, Filtered on Unfiltered" established a filter pore size of "nominally 1 micron" for filters that must be used in filtering groundwater samples for metals. This rule supersedes the pore size specifications listed in this document.

All groundwater samples for trace metals must use a 1.0 micron filter size, not the 0.45 micron filter discussed in this document.

Filtration for any other dissolved constituents (i.e., orthophosphorous, silica, etc.) still require the use of a 0.45 micron filter. Any analysis for dissolved metals in surface water sources must use the 0.45 micron filter.

Any questions concerning this change should be directed to the Quality Assurance Section, Florida Department of Environmental Protection (904) 488-2796.

Foreword
DER SOP
June 92
Page 1 of 1

FOREWORD

This document presents standard operating procedures for laboratory and field activities. These protocols may be incorporated by reference into the Comprehensive Quality Assurance Plans required by Chapter 17-160, F.A.C.

In addition, this document specifies the format, content and requirements of a Comprehensive Quality Assurance Plan that adopts these procedures.

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- A Definitions
- B Selected References

ACRONYMS

AOAC	Association of Official Analytical Chemists
ASTM	American Society for Testing and Materials
CCC	Continuing Calibration Check
CFR	Code of Federal Regulations
CL	Confidence Level
CLP	Contract Laboratory Program
CompQAP	Comprehensive Quality Assurance Plan
CV	Coefficient of Variation
DER	(State of Florida) Department of Environmental Regulation
DQO	Data Quality Objective
EPA	(United States) Environmental Protection Agency
FR	Federal Register
HRS	(Florida Department of) Health and Rehabilitative Services
I	Industrial Index
IDL	Instrument Detection Limit
MDL	Method Detection Limit
NIST	National Institute of Standards and Technology (previously NBS)
NPDES	National Pollutant Discharge Elimination System
NTIS	National Technical Information Service
OGC	Office of General Counsel (DER)
PEA	Performance Evaluation
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAP	Quality Assurance Plan
QAPP	Quality Assurance Project Plan
QAO	Quality Assurance Officer
QAS	Quality Assurance Section
QC	Quality Control
R	Recovery (%R: Percent R)
RPD	Relative Percent Difference
RQAP	Research Quality Assurance Plan
RSD	Relative Standard Deviation (%RSD: Percent RSD)
SD	Standard Deviation
SOP	Standard Operating Procedure
SRM	Standard Reference Material
UIC	Underground Injection Control
U.S.C.	United State Congress
USGS	United States Geological Survey
UST	Underground Storage Tank
VOC	Volatile Organic Compound
WMD	Water Management District

1.0 INTRODUCTION AND USE

Organizations who perform analysis and/or sampling activities for programs specified in Categories 2C, 3 or 4 of Chapter 17-160, F.A.C. are required to have an approved Comprehensive Quality Assurance Plan (CompQAP) on file with DER.

The Comprehensive Quality Assurance Plan may be submitted in one of three ways:

1. Prepare a document written according to the requirements specified in the "DER Manual for Preparing Quality Assurance Plans" (DER-QA-001/90);

2. Adopt and use all relevant Standard Operating Procedures (SOPs) discussed in Chapter 3 through 13 of this document by submitting a Statement of Intent with a list of equipment, capabilities and staff; or

3. Adopt selected SOPs and incorporate them into a document that is written in accordance with DER-QA-001/90.

If SOPs are adopted in a CompQAP, the Department requires and expects that all adopted protocols will be followed **WITHOUT MODIFICATIONS**, when collecting and/or analyzing samples related to DER rules and regulations.

The procedures in this document **SHALL NOT** be used as:

1. Substitution for Department-written Standard Operating Procedures that have been written for a specific program activity; or

2. Replacement or substitution of the Comprehensive Quality Assurance Plan and/or a Quality Assurance Project Plan (Category 2C, 3 and 4) requirement.

1.1 USING THIS MANUAL

1.1.1 Manual Organization

1. Chapter 2 of this document outlines the format, specifications and submission requirements for CompQAPs that adopt all or part of the Department's SOPs.

2. Chapters 3 through 13 discuss the specific standard operating procedures (SOPs) that may be incorporated by reference into a Comprehensive Quality Assurance Plan.

3. Appendix A of this document provides of list of terms AS THEY ARE DEFINED AND USED by the DER Quality Assurance Section and EPA.

4. Appendix B of this document provides of list of reference documents.

1.1.2 Mandatory and Recommended Requirements

The words "must", "shall", "may", and "should" indicate differences between mandatory and recommended requirements:

1. The words "must" and "shall" are used to indicate those topics and protocols that must be followed in order to be in compliance with the SOPs.

2. The words "may" and "should" indicate protocols or topics that the Department recommends following, but are not mandatory.

Some of the information in Chapters 3 through 13 of this document is included for informational and educational purposes. Such information is identified by double brackets ([[]]) which surround the text.

1.2 GENERAL SUBMITTAL REQUIREMENTS

1.2.1 Time of Submittal

1. All required Quality Assurance Plans must be submitted and approved or approved pending before sampling and analysis on the specified project can begin. Requirements from other DER sections (work plan, permit approval, consent order, etc.) may need to be satisfied before work can begin.

2. All consultants and laboratories are encouraged to submit plans well in advance of anticipated work. Most Quality Assurance Plans are returned for at least one revision before approval. If possible, plans should be submitted at least 60 days before the anticipated start of sampling and analysis activities.

1.2.2 Parties Responsible for Submitting Comprehensive QA Plans

1. The CompQAP must address the capabilities of the specified organization in a single document. CompQAPs for separate programs or purposes (i.e., Chemical, Biological, Hazardous Waste Sampling, "Tanks" work, etc.) are not accepted by DER.

2. If an organization or corporation has sampling and analysis capabilities in the same physical location, a single CompQAP must be submitted. Separate CompQAPs will not be accepted.

3. Organizations who: 1) have the same street or mailing address; 2) have identical key personnel; and 3) have identical equipment and capabilities shall not submit multiple CompQAPs for approval even if the name of the organization is different.

4. If required, all organizations shall perform sampling and/or analysis activities under the CompQAPs of their organization. Organizations shall not operate under the CompQAP of another organization.

5. If a corporation or company with multiple branch offices or laboratories wishes to submit Comprehensive QA Plans for approval, these guidelines must be followed:

a. Laboratory CompQAPs (includes full service laboratories with both field and laboratory capability)

1. University systems that require CompQAPs may submit a CompQAP for each Department or laboratory.

2. A separate CompQAP must be submitted for each laboratory located in different cities.

3. A single CompQAP for multiple (branch) laboratories of a corporation is acceptable if:

a. The branch laboratories operate under identical Standard Operating Procedures Manuals;

b. The branch laboratories are electronically linked with each other through a single Laboratory Information Management System which incorporates data from all branches and generates the Quality Assurance Targets for all laboratories;

c. All data from each laboratory is accessible through the laboratory information system to all other branches;

d. Employees from other branches are under direct supervision of the project manager in charge of the project, even if that manager is located in another branch office; and

e. The project manager is responsible for reviewing and approving final data reports even if the data is generated by another branch.

The CompQAP must include assertions that the above-stated requirements have been met (Section 1.0 of a CompQAP that has adopted all SOPs or Section 4.0 of a CompQAP that adopts some of the SOPs).

b. Field CompQAPs

1. A single Comprehensive QA Plan is acceptable for all branch offices of the company or corporation if each office has the same capabilities and if similar equipment and field procedures are used.

2. CompQAPs from individual branches shall be required if the capabilities, or standard operating procedures are not the same for all branch offices.

1.2.3 Amendments to Approved Comprehensive QA Plans

1. By rule, approval for a Comprehensive Quality Assurance Plan must be renewed once a year on the anniversary date of DER approval. If nothing has changed during the previous year, a letter must be sent stating that the laboratory or consulting firm wishes to renew approval.

- a. Failure to submit amendments or a letter will mean that approval of the plan will expire on the anniversary date.
 - b. All QA Project Plans which reference an expired CompQAP will be returned unreviewed. If the approval of the CompQAP is renewed at a later date, the unreviewed plans may be resubmitted.
 - c. If approval of a QA Plan has expired, the approval may be renewed by submission of the required amendments or renewal letter described above. Such approval will be from the date that the amendments are approved, not from the anniversary date.
2. In order to renew approval, the revisions (or letter) must be received by the QA Section 30 days prior to the anniversary date, and must be approved on the first or second submission. Amendments at any other time will not be reviewed unless:
- a. The company or laboratory changes name or organizational structure (i.e., buys or is bought by another organization).
 - b. The company or laboratory adds new capabilities (e.g., ability to sample hazardous waste) or new methods (e.g., pesticide analytical program is expanded) which will be used for work performed under the QA plan.
 - c. The company or laboratory acquires new instrumentation or equipment that will add new capabilities. New equipment or instrumentation that does not provide new capabilities (e.g., addition of another Atomic Absorption spectrophotometer for previously approved furnace work or replacement of an instrument) does not require an immediate amendment.
 - d. DER requests amendments to clarify, correct or update portions of the CompQAP.
3. When a section (or sections) is (are) amended, the revision date of the amended section(s) shall be updated to the current date.

2.0
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September 92
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2.0 INSTRUCTIONS FOR INCORPORATING STANDARD OPERATING PROCEDURES INTO COMPREHENSIVE QUALITY ASSURANCE PLANS

2.1 GENERAL

2.1.1 The protocols that are outlined in this document must be carefully reviewed before making the decision to adopt the SOPs

2.1.2 Organizations Adopting all SOPs

2.1.2.1 Organizations that adopt all SOPs that are relevant to their operations will be given approval for all analytical methods or sampling capabilities that are consistent with the submitted equipment and instrumentation lists.

2.1.2.2 Determination of approval status will occur within one month of submission.

2.1.2.3 Note that approval MAY NOT include all capabilities (i.e. analytical methods or sampling protocols) that are listed in the CompQAP. Approval will be contingent on the use of approved analytical methods (see 2.3.3.4.b), adoption of all appropriate SOPs, and use of appropriate equipment and/or instrumentation.

2.1.3 Organizations Adopting Portions of the SOPs

2.1.3.1 An organization that does not wish to adopt all SOPs pertinent to their operations must submit a full Comprehensive Quality Assurance Plan specified by DER-QA-001/90, however, the selected SOPs may be incorporated by reference.

2.1.3.2 Organizations that adopt all pertinent sample collection protocols may be given limited approval pending status subject to the requirements specified in 2.1.2 above.

2.2 STATEMENT OF INTENT TO COMPLY WITH THE DEPARTMENT OF ENVIRONMENTAL REGULATION STANDARD OPERATING PROCEDURES FOR LABORATORY OPERATIONS AND SAMPLE COLLECTION ACTIVITIES

A Statement of Intent is required when adopting all or portions of the protocols listed in this document.

1. The Statement is a two-part document which must be completed and signed by the appropriate parties.

2. The Statement MUST BE SUBMITTED WITH SUPPORTING INFORMATION. If adopting all SOPs, the organization must submit tables which include lists of personnel, methods and equipment

(Section 2.3). If adopting some of the SOPs, the organization must submit a CompQAP with the Statement (Section 2.4).

3. The document WITH ORIGINAL SIGNATURES must be submitted with all other required information; it will not be accepted if submitted without supporting documentation (see 2.3 and 2.4) or if a copy of facsimile is submitted.

4. This document may be obtained from the a DER District Office or by writing to the Florida Department of Environmental Regulation, DER Library, 2600 Blair Stone Road, Tallahassee, Florida 32399-2400.

5. This document shall not be retyped or modified. A Statement of Intent that has been retyped by the organization WILL NOT BE ACCEPTED.

2.2.1 Part I - List of SOPs

2.2.1.1 The first part is a list of all SOP activities and protocols. Complete this portion by:

- a. Filling in the name and address of the organization and the CompQAP number (if already assigned);
- b. Placing an "X" or checkmark by each of the SOPs that your organization intends to adopt. If the organization does not have the capability, personnel or equipment to perform certain tasks (i.e. hazardous waste sample collection, wastewater sample collection, etc.), DO NOT check the associated SOPs.

2.2.1.2 Mandatory SOPs that must be Adopted

- a. If an organization intends to follow only adopted SOPs, Table 2.1 identifies the document chapters and/or sections that must be adopted and checked on the Statement of Intent.
- b. Organizations that adopt portions of the SOPs must indicate those that are to be adopted. NOTE: THE INFORMATION THAT IS NOT ADOPTED BY SOPs MUST BE DISCUSSED IN THE TEXT OF THE CompQAP (see Section 2.4).

2.2.1.3 Before determining the status of your QA Plan, this part of the statement will be checked for inconsistencies and/or omission of pertinent SOPs as well as correlation between the SOPs and the submitted equipment and instrument lists.

2.2.2 Part II - Certification

2.2.2.1 The second part of this document is a certification. This portion certifies that the organization:

TABLE 2.1
MANDATORY SOP CHAPTERS TO BE ADOPTED

CHAPTER/SECTION TITLE	LABORATORY ONLY	FIELD (SAMPLE COLLECTION ACTIVITIES)	FULL SERVICE FIELD & LAB
CHAPTER 3 - ORGANIZATION AND RESPONSIBILITY	X	X	X
CHAPTER 4 - SAMPLING PROCEDURES			
Section 4.0 - General		X	X
Section 4.1 - Equipment Decontamination			
Section 4.1.1 General Concerns	X (1)	X	X
Section 4.1.2 Reagents	X (1)	X	X
Section 4.1.3 General Decontamination Protocols	X (1)	X	X
Section 4.1.4 Sampling Equipment	X (1,2)	X	X
Section 4.1.5 Wastewater Automatic Samplers	X (1,2)	X (2)	X (2)
Section 4.1.6 Field Filtration Equipment	X (1,2)	X (2)	X (2)
Section 4.1.7.1 Teflon Tubing	X (1,2)	X (2)	X (2)
Section 4.1.7.2 - 5 Non-teflon Tubing	X (1,2)	X (2)	X (2)
Section 4.1.8 Pumps used for Purging and/or Sampling	X (1,2)	X (2)	X (2)
Section 4.1.9 Field Meters, Flow Meters and other Field Instruments	X (1,2)	X (2)	X (2)
Section 4.1.9 Non-Sampling Equipment	X (1,2)	X (2)	X (2)
Section 4.1.10 Analyte-Free Water Containers	X (1,2)	X (2)	X (2)
Section 4.1.11 Ice Chests and Shipping Containers	X (1,2)	X (2)	X (2)
Section 4.2 Aqueous Sampling Procedures			
Section 4.2.1 General Sampling Concerns		X (3)	X (3)
Section 4.2.2 Special Sample Handling Procedures		X (3)	X (3)
Section 4.2.3 Surface Water		X (4)	X (4)
Section 4.2.4 Wastewater		X (4)	X (4)
Section 4.2.5 Groundwater		X (4)	X (4)
Section 4.2.6 Wells with In-Place Plumbing		X (4)	X (4)
Section 4.2.7 Potable Wells Sampling		X (4)	X (4)
Section 4.2.8 Drinking Water Supply Systems		X (4)	X (4)
Section 4.2.9 Temporary Well Points		X (4)	X (4)
Section 4.2.10 Air Stripper and Remedial Treatment Systems		X (4)	X (4)
Section 4.2.11 Bioassay		X (4)	X (4)
Section 4.3 Solid Matrix Sampling			
Section 4.3.1 General Concerns		X (3)	X (3)
Section 4.3.2 Sample Handling Protocols after Sample Acquisition		X (3)	X (3)
Section 4.3.3 Composite Soil Samples		X (3)	X (3)
Section 4.3.4 Soil Sampling		X (4)	X (4)
Section 4.3.5 Sediment Sampling		X (4)	X (4)

TABLE 2.1, continued
MANDATORY SOP CHAPTERS TO BE ADOPTED

CHAPTER/SECTION TITLE	LABORATORY ONLY	FIELD (SAMPLE COLLECTION ACTIVITIES)	FULL SERVICE FIELD & LAB
Section 4.3.6 Fish Tissue Sampling		X (4)	X (4)
Section 4.3.7 Shellfish Sampling		X (4)	X (4)
Section 4.3.8 Domestic Wastewater Residual Sampling		X (4)	X (4)
Section 4.3.9 Hazardous Waste Sampling		X (4)	X (4)
Section 4.3.10 Macrobenthic Invertebrates		X (4)	X (4)
Section 4.4 Sampling Handling			
Section 4.4.1 Sampling Container Cleaning	X (5)	X	X
Section 4.4.2 Sample preservation, container types and holding times	X	X	X
Sources of preservation	X (6)	X (6)	X (6)
Section 4.4.3 Sample Dispatch		X	X
Section 4.4.4 Field Reagent Handling		X	X
Section 4.4.5 Field Waste Disposal		X	X
CHAPTER 5 - SAMPLE CUSTODY AND DOCUMENTATION			
Section 5.1 General Requirements for Custody and Documentation	X	X	X
Section 5.2 Preparation of Field Sampling Supplies and Equipment	X (1,5)	X	X
Section 5.3 Custody and Documentation Requirements for Field Operations		X	X
Section 5.4 Custody and Documentation Requirements for Laboratory Operations	X		X
Section 5.5 Electronic Data Documentation	X (4)	X (4)	X (4)
Section 5.6 Legal or Evidentiary Custody Procedures	X (4)	X (4)	X (4)
CHAPTER 6 - ANALYTICAL PROCEDURES	X		X
CHAPTER 7 - CALIBRATION PROCEDURES AND FREQUENCY			
Section 7.1 Introduction	X	X	X
Section 7.2 General Considerations	X	X	X
Section 7.3 Standard Receipt and Traceability	X	X	X
Section 7.4 Frequency of Standard Preparation and Standard Storage	X	X	X
Section 7.5 Minimum Calibration Requirements for Field Instruments		X	X
Section 7.6 Laboratory Instruments	X		X
Section 7.7 Support Equipment Calibration	X		X
Section 7.8 Calibration Documentation	X	X	X
Section 7.9 Definitions	X	X	X

TABLE 2.1
MANDATORY SOP CHAPTERS TO BE ADOPTED

CHAPTER/SECTION TITLE	LABORATORY ONLY	FIELD (SAMPLE COLLECTION ACTIVITIES)	FULL SERVICE FIELD & LAB
CHAPTER 8 - PREVENTATIVE MAINTENANCE	X	X	X
CHAPTER 9 - MINIMUM QC REQUIREMENTS AND ROUTINES TO CALCULATE AND ASSESS PRECISION, ACCURACY AND METHOD DETECTION LIMITS			
Section 9.1.1 Minimum Field Quality Control Requirements		X	X
Section 9.1.2 Minimum Laboratory Quality Control Requirements	X		X
Section 9.2 Routine Methods used to Assess Precision and Accuracy	X		X
Section 9.3 Method Detection Limits and Practical Quantitation Limits	X		X
Section 9.4 Documentation	X	X	X
CHAPTER 10 - DATA REDUCTION, VALIDATION AND REPORTING	X	X	X
CHAPTER 11 - CORRECTIVE ACTION	X	X	X
CHAPTER 12 - PERFORMANCE AND SYSTEMS AUDITS	X	X	X
CHAPTER 13 - QUALITY ASSURANCE REPORTS	X	X	X

X - Section must be adopted, subject to the following comments:

- (1) Laboratories providing equipment cleaning services must adopt this section
- (2) Adopt only those protocols for which equipment is provided (laboratories) and/or listed (field and full service)
- (3) Mandatory if protocols discussed in the subsection are adopted
- (4) Adopt only if applicable
- (5) Laboratories providing sample containers must adopt this section
- (6) The manner in which preservatives are provided/obtained must be specified

- a. Has obtained copies of all documents specified by the SOPs;
- b. Intends to follow all SOPs noted in Part I;
- c. Has the equipment and capability to perform the protocols specified by Part I; and
- d. Is committed to generating data of a known and verifiable quality.

2.2.2.2 The Statement of Intent must be signed by at least two individuals:

- a. An individual who will be ultimately responsible for the quality, and reliability of all data generated by the organization (usually the laboratory manager, director of field operations, etc.); and
- b. The Quality Assurance Officer(s) of the organization.

2.2.2.3 The Statement must be properly dated and signed.

2.3 COMPQAP FORMAT WHEN ALL SOPS ARE ADOPTED

Organizations adopting the SOPs that are pertinent to their operations are required to submit the following information:

2.3.1 Laboratory or Full Service Organization

1. Statement of Intent;
2. Names of the key personnel within the organization;
3. Table of Method numbers, matrix identification and QA Objectives for Precision and Accuracy;
4. List of all analytical instrumentation and support equipment.
5. List of sampling capabilities (if applicable); and
6. List of sampling equipment (if applicable).

2.3.2 Sample Collection (Field Activities)

1. Statement of Intent;
2. Names of the key personnel within the organization;
3. Table of Method numbers for field measurements;
4. List of sampling capabilities; and
5. List of sampling equipment.

2.3.3 Submission Format and Requirements

2.3.3.1 General Requirements

- a. All Comprehensive QA Plans shall be submitted to the QA section in 3-ring loose leaf binders. This allows the QA section and the consultant to easily update or amend specific pages or sections by removing outdated sections and inserting the new revisions.

b. All plans must be submitted as final plans. Draft documents or documents identified as draft will be returned unreviewed.

c. Incomplete plans or plans that do not conform to the formats specified in this document shall be returned unreviewed.

d. Only one (1) copy of the plan must be submitted to the DER QA Section. Note: DER program managers may request additional copies of the approved QA Plan for their records, however, only one plan should be submitted to the QA section.

e. The QA Plan must be submitted in numbered sections which correspond to the list of topics outlined in Section 2.3.1 or 2.3.2 above. All sections (except the Statement of Intent) must be identified with a document control header which shall be placed in the upper corner opposite the binding of each document page with the following information:

1. Section No. - identifies the Section/Element
2. Date - is the date of the revision
3. Page ____ of ____ - identifies the specific page within in the section, and the total number of pages in the section (see Fig. 2.1).

NOTE: SECTIONS ARE TO BE NUMBERED ACCORDING TO TITLES IN SECTIONS 2.3.3.3 THROUGH 2.3.3.6 OF THIS DOCUMENT.

2.3.3.2 Statement of Intent - Complete per instructions in Section 2.2 above.

2.3.3.3 Section 1.0 - Key Personnel

- a. Provide a list of the salaried employees with title.
- b. The list should include **KEY PERSONNEL**, only. Key personnel are defined as: all individuals from the owner/director/manager through line supervisors.
- c. Provide a list of branch offices (including address and phone number) **IF** this CompQAP is to be used by more than one office. Key personnel (with title) for each office shall be identified

2.3.3.4 Section 2.0 - Organization Abilities (Methods, Matrices, QA Targets and Sampling Capabilities)

This section must outline the capabilities of the organization in terms of analytical or testing methods (this section) **AND, IF APPLICABLE**, sampling capabilities (Section 2.3.3.5). All information shall be presented in table form. The number of tables that will be required are dependent on the capabilities of the organization and are outlined on Table 2.2.

The instructions in this section pertain to the required tables for analytical and field measurement capabilities:

Fig. 2.1

DOCUMENT CONTROL HEADER PLACEMENT

Section No. _____
Date: _____
Page _____ of _____

This diagram illustrates the placement of document control headers in the top right corner of a page. The header information is aligned to the right. On the left side of the page, there are two circles representing binder holes.

Section No. _____
Date: _____
Page _____ of _____

This diagram illustrates the placement of document control headers in the top left corner of a page. The header information is aligned to the left. On the right side of the page, there are two circles representing binder holes.

Table 2.2

REQUIRED TABLES IN SECTION 2.0 OF COMPOAPS THAT ADOPT ALL SOPs

<u>Table Identification</u>	<u>Laboratory</u>	<u>Field</u>	<u>Full-Service</u>
Analytical Test Methods	X		X
Sample Preparation Methods *	X		X
Field Measurements		X	X
Sampling Capabilities		X	X

* Required only if SW 846 methods are proposed as analytical test methods.

Laboratory - Provides only analytical services
 Field - Provides only sample collection services
 Full-Service - Provides both sample collection and analytical services

a. General Requirements

1. This section must include ALL field, biological and chemical analytical (measurement) capabilities of a laboratory or consulting firm that are pertinent to DER programs and rules. The components in each method must be listed separately.

2. This information must be presented in table form as described in Section 2.3.3.4.c below.

3. The analytical methods for field measurement parameters (pH, specific conductance, temperature, etc.) must be presented as a separate table. If equipment such as OVAs are used to screen samples in the field, the method should not be listed in this section. The equipment must be listed on the list of field equipment (Section 2.3.3.6.a) and pertinent calibration and preventative maintenance SOPs must be checked on the Statement of Intent.

4. Precision, Accuracy and Detection limits or goals must be provided for all parameters (EXCEPT field measurements and screening methods). If not available from in-house data, literature or method values may be used but must be identified with appropriate annotations. DER encourages generation of in-house QA objectives for all components (unless otherwise specified by the method) and expects that this table will be completed with in-house data no later than one year after final approval.

5. The precision, accuracy and detection limits presented in the CompQAP shall be the routine target values used by the consultant. Deviations from these criteria must be addressed in quality assurance project plans.

b. Approved Methods

1. All analyses must be performed in accordance with currently accepted DER methods (listed on Tables 2.3, 2.4 and 2.5).

2. If there is no DER approved method for a specified component, the following alternatives may be used:

a. DER will review proposed existing methods other than those listed in Tables 2.3, 2.4 and 2.5 and approve the use of the method, if appropriate. Sources of such methods may be (see references in Appendix B for specific revision dates):

1. Standard Methods
2. ASTM - American Society for Testing Materials
3. USGS - U.S. Geological Survey
4. AOAC - Methods of Analysis of the Association of Official Analytical Chemists

b. If no appropriate method exists in any of the above-referenced documents, a full method validation

TABLE 2.3

**DER APPROVED ANALYTICAL METHODS AND REFERENCES FOR WATER
AND BIOLOGICAL ANALYSES**

DRINKING WATER

These methods must be used when analyzing potable waters for compliance with Chapters 17-550, 17-551, 17-555 and 17-560, F.A.C. and/or for certification under the HRS Drinking Water Certification program.

1) 40 CFR Part 141, National Primary Drinking Water Regulations, July 1, 1991 Edition, Code of Federal Regulations, Subpart C (Monitoring and Analytical Requirements, sections 141.21 to 141.30) and Subpart I (Control of Lead and Copper, section 141.89).

2) "Methods for the Determination of Organic Compounds in Drinking Water," EPA 600/4-88-039, December 1988.

3) Chapter 10D-41, sections 50 to 62, F.A.C., Amended 11-15-90, HRS Laboratory Certification Rules (Drinking Water Analyses).

4) "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, revised March 1983.

5) Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 17th Edition, 1989.

6) "Manual for Certification of Laboratories Analyzing Drinking Water, Criteria and Standards Quality Assurance" EPA 570/9-90-008, April 1990 as updated by Change I (EPA 570/9-90-008A), October 1991 and Change 2 (EPA-814B-92-002), September 1992.

7) 40 CFR Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, July 1, 1991, Appendix A.

NOTES:

1) "500" series methods shall be used only for regulatory analysis of drinking water (see above statement) unless approved by DER for use in other matrices or programs.

2) Only Methods 502.1, 502.2, 503.1, 504, 505, 507, 508, 508A, 515.1, 524.1, 524.2 and 531.1 from Reference 2 and Methods 604, 606, 609, 612, 613 and 625 from Reference 7 are approved for analysis of drinking water by state certified laboratories.

**SURFACE WATER, GROUNDWATER, AND WASTEWATER (DOMESTIC/INDUSTRIAL)
EFFLUENTS**

1) 40 CFR Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Tables IA, IB, IC, ID, and IE, as published in the Federal Register, Vol. 65, No. 165, pp. 50758-50770, October 8, 1991.

2) Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, revised March 1983.

TABLE 2.3, continued

3) "Test Methods for Evaluating Solid Waste, Physical Chemical Methods", Third Edition (EPA SW-846), 1986 as amended by Final Update 1, November 1990.

4) 40 CFR Part 261, Identification and Listing of Hazardous Waste, July 1, 1991, Appendix III (Chemical Analysis Test Methods).

NOTES:

1) Laboratories analyzing samples in support of NPDES Permits are limited to methods specified in Reference 1 above or those specifically approved for use by EPA.

2) Only those methods in Reference 2 which are specified in Reference 1 are approved.

3) Methods and protocols specified by References 3 and 4 above must be used when analyzing samples in support of RCRA related activities.

Approved Methods to be used in support of DER Rules and Programs:

1) Pesticides and Herbicides:

a. "Methods for the Determination of Nonconventional Pesticides in Municipal and Industrial Wastewater," EPA 821 RR-92-002, April 1992. Note: methods in the Appendix (EV-024 and EV-025) require method validation from the laboratory.

b. N-Methyl Carboxylamines and N-Methyl Carbamates - EPA Method 531 (600/4-88-039). Approved ONLY for the analysis of these components in groundwater samples.

2) Inorganics:**

a. BROMINE - SM 408E, SM 4500-Cl-G - The approved method is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e. negligible interference).

b. BROMATES - EPA 300.0 B - "Determination of Inorganic Anions in Water by Ion Chromatography" by Jack D. Pfaff, Carol A. Brockoff, and James W. O'Dell, U.S. EPA, Cincinnati, Ohio, 45268.

c. CHLOROPHYLLS - SM 1002G, SM 10200 H

d. CORROSIVITY (CaCO₃ Stability, Langelier Index):

1) SM 203, SM 2330

2) ASTM D513-82

e. EDB in groundwater:

1) EPA 601 modified using an electron capture detector instead of an electrolytic conductivity detector

2) EPA 504

TABLE 2.3, continued

- 2) Inorganics, continued
 - e. EDB in groundwater, continued:
 - 3) HRS (Florida Department of Health and Rehabilitative Services) Method for the analysis of EDB
 - 4) EPA 8011
 - f. ODOR - SM 207, SM 2150
 - g. SALINITY:
 - 1) SM 210A, SM 2520 B
 - 2) SM 210B, SM 2520 C
 - 3) SM 210C
 - h. TASTE:
 - 1) SM 211 A, B, SM 2160 B, C, D
 - 2) ASTM 1292-86
 - i. TOTAL DISSOLVED GASES - SM 2810
 - j. TRANSPARENCY - 17-3.031(6), F.A.C.
 - k. UN-IONIZED AMMONIA - DER-SOP - DER Central Analytical Laboratory, Tallahassee, FL, Revision No. 1, October 3, 1983.
(Available from the DER QA Section)

** Methods coded SM XXX are from Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1983, those coded SM XXXX are from the 17th Edition, 1989 (except Chlorophyll where SM 1002G is from the 16th Edition and SM 10200H is from the 17th Edition). Methods coded ASTM are from the American Society for Testing Materials.

BIOLOGICAL

Microbiological

- 1) Drinking Water Analyses - 40 CFR Part 141, Subpart C (Monitoring and Analytical Requirements, section 141.21), July 1, 1991.
- 2) Water and Wastewater Analyses - 40 CFR Part 136, Table IA as published in the Federal Register, Vol. 65, No. 165, pp. 50758-50770, October 8, 1991.
- 3) "Microbiological Methods for Monitoring the Environment" EPA 600/8-78-017, 1978.
- 4) Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 17th Edition, 1989.

Bioassay

- 1) "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (Fourth Edition)" EPA 600/4-90-027, September, 1991.
- 2) "Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms (Third Edition)" EPA 600/4-91-002, 1991.

TABLE 2.3, continued

3) "Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms (Second Edition)" EPA 600/4-91/003, 1991.

Macrobenthic Identification and Enumeration

1) "Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Waters", ORD, Washington, D.C., November 1990

2) Standard Methods for the Examination of Water and Wastewater, Part 10500, 17th Edition, APHA, 1989.

TABLE 2.4

**DER APPROVED ANALYTICAL METHODS AND REFERENCES FOR
SEDIMENTS, SOILS, RESIDUALS AND SOLID AND HAZARDOUS WASTES**

SOILS AND SEDIMENTS

- 1) "Test Methods for Evaluating Solid Waste, Physical Chemical Methods", Third Edition (EPA SW-846), 1986 as amended by Final Update 1, November 1990.
- 2) "Procedures for Handling and Chemical Analysis of Sediments and Water Samples" EPA/Corps of Engineers, EPA/CE-81-1, 1981.
- 3) *"USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis", ILMO 2.0 (July 1990) and ILMO 2.1 (September 1991).
- 4) *"USEPA Contract Laboratory Program Statement of Work for Organic Analysis", ILMO 2.0 (July 1990) and ILMO 2.1 (September 1991).
- 5) Estuarine Sample Preparation and Analysis - Deepwater Ports Maintenance Dredging and Disposal Manual, Department of Environmental Regulation, Coastal Zone Management, Revision 4, December 1984.

DOMESTIC AND INDUSTRIAL SLUDGES (RESIDUALS)

- 1) "Test Methods for Evaluating Solid Waste, Physical Chemical Methods", Third Edition (EPA SW-846), 1986 as amended by Final Update 1, November 1990.
- 2) "POTW Sludge Sampling and Analysis Guidance Document" USEPA Permits Division, August 1989.

SOLID AND HAZARDOUS WASTES

- 1) "Test Methods for Evaluating Solid Waste, Physical Chemical Methods", Third Edition (EPA SW-846), 1986 as amended by Final Update 1, November 1990.
- 2) 40 CFR Part 261, Identification and Listing of Hazardous Waste, July 1, 1991, Appendix III (Chemical Analysis Test Methods).
- 3) *"USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis", ILMO 2.0 (July 1990) and ILMO 2.1 (September 1991).
- 4) *"USEPA Contract Laboratory Program Statement of Work for Organic Analysis", ILMO 2.0 (July 1990) and ILMO 2.1 (September 1991).

* Methods from these references shall be used and/or referenced by laboratories under direct contract to EPA to perform analyses for Superfund (CERCLA) site investigations.

TABLE 2.5

DER APPROVED METHODS FOR SPECIAL CONDITIONS
(INCLUDES DRAFT AND MODIFIED METHODS)

SOLID SAMPLES (Soils, Sediments, Sludges, Residuals, etc.):

- 1) Total Recoverable Hydrocarbons (Petroleum) - SW 846 method 9073 (draft)*
 - 2) Total Halides:
 - a) SW 846 method 5050/9056
 - b) SW 846 method 5050/9252
 - c) SW 846 method 5050/9253
- * Copy available from the DER QA Section

MODIFIED METHODS:

EPA Methods 300.0 (Revised August 1991) and 9056 - Method 300.0 may be used for the analysis of the METHOD SPECIFIED ions in groundwater and surface water. Method 9056 may be used for the METHOD SPECIFIED ion EXCEPT for fluoride.

EPA Methods 601, 602, 624 and 625 - Capillary columns may be used instead of the specified packed columns if the laboratory can meet the performance criteria (precision, accuracy and method detection limits) of the method.

EPA Methods 601 & 602 - The photoionization detector and electrolytic conductivity detector may be used in series if the laboratory can meet the performance criteria of the specified methods.

EPA Methods 602, 8020, & 8021 - May include the analyses for xylenes and methyl tert-butyl ether (MTBE). Note: required for Chapter 17-770 work.

EPA Method 610, 625, 8100, 8310, 8250 & 8270 - May include the analyses for 1-methylnaphthalene and 2-methylnaphthalene. Note: required for Chapter 17-770 work.

EPA Method 5030/8010 - Must be modified to analyze for EDB in soils. An electron capture detector instead of a electrolytic conductivity detector must be used.

package (as outlined in Rule 17-160.520, F.A.C.) must be submitted as an appendix to the CompQAP.

3. Modified or Alternative Methods

a. Modified Methods - Any modification to approved analytical methods must be identified in the table as "mod", and details of the modification must be provided as an attachment even if the modification has been approved by DER or EPA.

b. Alternative methods - A laboratory who wishes to use an alternative method that is not listed on Tables 2.3, 2.4 or 2.5 must list the method number in the table, and added as an attached a method validation/equivalency package that has been prepared per the instructions in Appendix E of DER-QA-001/90.

c. If components which are not specified by the analytical method are analyzed (e.g. malathion using method 608), a method validation package (see Appendix E of DER-QA-001/90) must be submitted for review and approval, and must be included as an appendix to the CompQAP.

ALL METHODS THAT DO NOT HAVE EPA OR DER APPROVAL MUST BE REVIEWED AND APPROVED BY DER BEFORE USING ON ANY DER-RELATED WORK.

c. Methods Section Format - Laboratories must include a minimum of two tables (sample preparation and analysis methods); three if sample collection activities are included in capabilities. Field QA Plans must include a table of analysis methods.

1. Sample Preparation - Sample preparation method numbers must be presented for any EPA SW 846 method that does not specify a sample preparation (extraction, digestion, clean up, etc.) method. These sample preparation methods shall identify the sample preparation number and the appropriate methods to which the procedure applies (see Fig. 2.2).

2. Analytical methods - This table must include information concerning analytes, method numbers, matrices and quality control criteria:

a. Method Number

1. Method numbers must be provided for each proposed matrix and component.

2. List all equivalent methods (i.e. SW 846 and "600" series methods) if seeking approval to perform analyses by both methods.

Fig. 2.2

EXAMPLE OF SAMPLE PREPARATION TABLE

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Page 5 of 35

Table 2.1
SAMPLE PREPARATION METHODS

Sample Prep. Method Number	Description	Matrix	Sample Prep. for these Methods:
3005	Acid Digestion	Water	4010, 7020, 7040, 7080, 7130
3020	Acid Digestion	Water, soil	7041, 7091, 7131
3510	Liquid/Liquid Extraction	Water	8080, 8140
3610	Alumina Cleanup	Water, soil	8080, 8140
3640	GPC Cleanup	Soil, tissues	8080, 8140

b. Matrix Identification - Since precision and accuracy objectives are matrix specific, each method must identify the matrices to be analyzed. Identify the matrix groups as follows:

1. Drinking water
2. Water - Includes surface water, groundwater and wastewater (these may be identified separately)
3. Saline waters
4. Soils and sediments
5. Chemical wastes (includes sludges and residuals from domestic or industrial wastewater processing)
6. Biological tissues (includes shellfish, fish and mammalian)

c. Component Name - All components (compounds or chemicals) that are to be analyzed must be listed.

1. If a primary method (ex. Method 608) has published addenda (ex. Method 608.1 and Method 608.2) and if the components in the addenda are to be analyzed, these analytes may be included under the same method number heading.

2. Use component names as they are listed in the method.

d. Precision Targets

1. Must be described by the maximum allowable variance and reported as the upper acceptable value (e.g. 5%). The specific units (i.e. % RSD, RPD, I) shall be identified and calculated according to the formulas specified in Chapter 9 of this document.

NOTE: RPD AND THE INDUSTRIAL STATISTIC WILL BE CONVERTED TO % RSD IN THE QA DATA BASE.

2. All precision limits must be generated from in-house data. If no data exists, published method values or internal goals may be used until in-house values can be generated.

3. All internal goals, literature or method reference data must be identified.

e. Accuracy Targets

1. Must be reported as a range (e.g. 90-125%) and calculated per instructions in Chapter 9.

2. Accuracy target values should be derived from historical in-house data. If unavailable, method targets or internal goals may be used until in-house values are available.

3. All internal goals, literature or method targets must be properly identified.

f. Method Detection Limits (MDLs) or Practical Quantitation Limits (POLs)

1. The method detection limit or practical quantitation limit for each component must be provided.
2. Identify the reporting units (e.g. mg/kg, ug/L, etc.) and whether MDL or PQL.
3. Do not mix MDL and PQLs on the same tables.
Enter values for only one of the two types.
4. Specify the method by which the MDLs were calculated (see Chapter 9): EPA, IUPAC or USATHAMA.
 - a. Note: Since PQLs are based on the determination of MDLs, the method by which MDLs are calculated is required EVEN IF PQLs are reported.
 - b. The QA Data Base will convert all reported PQL values to MDLs using the definition of PQL specified in Chapter 9. This means that all PQL values will be divided by a factor of 4.
5. Criteria or Action level goals may be listed in lieu of MDLs or PQLs if these requirements are met:
 - a. Criteria or action levels that must be met for a specified permit or program exceed the minimum detection limit of the method by two orders of magnitude (e.g. MDL is 2 ppb, the criteria or required reporting level is 200 ppb).
 - b. These targets are identified by specific reference to Department Criteria or permit requirements.
 - c. The use of elevated targets is justified in this section.
- g. QA Target Concentration Ranges
 1. For each QA Target of precision and accuracy, identify the concentration level that was used to determine the value. The following codes and concentration definitions shall be used:
 - a. L - low range is the lower 20% of the linear calibration range.
 - b. M - mid range is defined as the concentrations from 20% to 80% in the linear calibration range.
 - c. H - high range is defined as the concentrations which are in the upper 80% of the linear calibration range.
 - h. The following acronyms shall be used to prepare these tables:
 1. EPA - refers to methods found in any EPA approved source

- 2. SM - refers to methods found in Standards Methods (specify edition)
- 3. ASTM - refers to method references from ASTM
- 4. USGS - refers to method references from the US Geological Survey documents
- 5. AOAC - refers to method references from the Methods of Analysis of the Association of Official Analytical Chemists
- 6. NA - Not applicable
- 7. MDL - Method Detection Limit
- 8. PQL - Practical Quantitation Limit
- 9. MOD - Modified Method
- 10. DW - Drinking water
- 11. SW - Surface water
- 12. GW - Groundwater
- 13. SED - Sediments (includes domestic sludges)
- 14. S - Soils
- 15. EFF - Effluent
- 16. HW - Hazardous Wastes (includes chemical wastes and sludges)
- 17. BIO - Samples from biological matrices (tissues, muscle, shellfish, etc.)
- 18. SA - Saline waters

d. Examples of the required formats for laboratory and field parameters are found in Figures 2.3 and 2.4.

2.3.3.5 Section 2.0 - Organization Abilities - Sampling Capabilities

Organization that analyze samples must include this list as an additional list in Section 2.0.

a. Sampling capabilities must be listed in table form by major matrix groups and major analyte groups. The matrix groups shall be identified as: groundwater, surface water, drinking water, wastewater, stormwater runoff, soils, sediments, tissues, shellfish, domestic wastewater sludges, hazardous waste sludges, and liquid hazardous wastes.

The major analyte groups are defined as follows:

- 1. VOCs - Volatile Organic Compounds to include Methods 601, 602, 624, 502.1 and 2, 503, 504, 524.1 and .2, 8010, 8020, 8021, 8240, 8260, EDB and purgeable organic halides.
- 2. Extractable Organics - To include base neutral components (e.g. polynuclear aromatics), acid extractable components (e.g. phenols), pesticides, herbicides, and PCBs.
- 3. Metals - All metallic analytes

EXAMPLE OF QA OBJECTIVES TABLE OF LABORATORY MEASUREMENTS

Fig. 2.3

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TABLE 005
QUALITY ASSURANCE OBJECTIVES

Method No.	Matrix	Analyte/Component	Precision % RSD	Conc. Range	Accuracy SD	Conc. Range	MDL ¹ µg/L / ng/kg
EPA 8060	Sed	Bis(2-ethylhexyl)phthalate	30	L	29 - 137	L	678
		Butyl benzyl phthalate	30	L	0 - 140	L	330
		Di-n-butyl phthalate	30	L	0 - 111	L	330
		Diethyl phthalate	30	L	0 - 100	L	330
		Dimethyl phthalate	30	L	0 - 100	L	330
		Di-n-octyl phthalate	30	L	19 - 132	L	330
EPA 8060 608	SW	Bis(2-ethylhexyl)phthalate	30	L	54 - 180	L	3
		Butyl benzyl phthalate	30	L	68 - 108	L	3
		Di-n-butyl phthalate	30	L	78 - 114	L	3
		Diethyl phthalate	30	L	36 - 54	L	3
		Dimethyl phthalate	30	L	54 - 90	L	3
		Di-n-octyl phthalate	30	L	66 - 84	L	3
EPA 8000 8001 608 608.1 608.2	SW SW SW EFF	Aldrin	35	L	58 - 100	L	0.01
		α-BHC	35	L	59 - 100	L	0.01
		β-BHC	35	L	61 - 100	L	0.01
		γ-BHC	35	L	61 - 100	L	0.01
		δ-BHC	35	L	60 - 100	L	0.01
		γ-BHC	35	L	45 - 119	L	0.2
		chlordane	35	L	55 - 115	L	0.02
		p,p'-DDD	35	L	50 - 100	L	0.02
		p,p'-DDE	35	L	60 - 120	L	0.05
		p,p'-DDT	35	L	60 - 120	L	0.05
		dieldrin	35	L	60 - 100	L	0.05
		endosulfan I	35	L	45 - 100	L	0.01
		endosulfan II	35	L	60 - 100	L	0.05
		endosulfan sulfate	35	L	30 - 147	L	0.01
		endrin	35	L	50 - 100	L	0.01
		endrin aldehyde	35	L	60 - 110	L	0.01
		heptachlor	35	L	60 - 110	L	0.2
		methoxychlor	35	L	50 - 100	L	0.05
		PCNB*	35	L	41 - 126	L	0.5
		toxaphene	35	L			

¹ MDLs have been calculated based on the IUPAC method of determining method detection limits.

-- BA Targets derived from literature values
* Additional component. Method validation study attached as appendix A

Fig. 2.4

EXAMPLE OF METHODS TABLE FOR FIELD MEASUREMENTS

TABLE XYZ			Section 2.0
QUALITY ASSURANCE OBJECTIVES			8-17-90
Field Measurements			Page 5 of 27
Method No.	Matrix	Analyte/Component	
EPA 150.1	W	pH	
EPA 170.1	W	Temperature	
SM 205	W	Specific Conductance	
EPA 360.2	W	Dissolved Oxygen	

4. Inorganic Anions - To include inorganic anions and other non-metallic tests: bromide; bromine; chloride; chlorine; iodide; nutrients (ammonia, Kjeldahl nitrogen, nitrate, nitrite, phosphate, o-phosphate); sulfate; silica; sulfite; acidity; alkalinity; dissolved oxygen and dissolved silica.

5. Organics - To include biochemical oxygen demand, chemical oxygen demand, oil and grease, total organic carbon, total recoverable petroleum hydrocarbons, phenolics, and surfactants.

6. Physical Properties - To include color, specific conductance, hardness, odor, pH, all residues (filterable, non-filterable, total, volatile and settleable), temperature and turbidity.

7. Microbiology - To include coliforms, streptococcus, enterococcus and other bacteria.

8. Other - Tests that must be included are: cyanide; biotoxicity; macroinvertebrate identification; and radionuclides.

See Figure 2.5 for the specified format.

2.3.3.6 Section 3.0 - Sampling Equipment and/or Analytical Instrumentation

On SEPARATE LISTS, list the laboratory and/or field equipment that is used by the organization:

a. Field Equipment

1. The sampling equipment list must be limited to equipment that the organization either owns or rents on a routine basis.

2. The list must be presented in format specified in Fig. 2.6. Note that construction materials of all purging and sampling equipment (including tubing) must be specified.

3. In addition to sample collection/purging equipment, the following must be listed:

- a. All field measurement equipment (pH meters, conductivity meters, etc.) and all field screening equipment (OVAs, GCs, etc.);
- b. All field decontamination equipment; and
- c. Miscellaneous and ancillary equipment (water level indicators, buckets, gloves, etc.).

b. Laboratory Analytical Instrumentation and Support Equipment

1. The list of laboratory instrumentation must identify the type of instrument. This list shall include but is not limited to:

Fig. 2.5

EXAMPLE OF TABLE OF SAMPLING CAPABILITIES

Section 2.0
6-27-91
Page 1 of 10

SAMPLING CAPABILITIES

<u>Parameter Group</u>	<u>Sample Source</u>
Extractable organics	Drinking water, surface water, groundwater, sediments, soils, hazardous waste sludges
Volatile organics	Drinking water, surface water, groundwater, sediments, soils, hazardous waste sludges
Trace Metals	Drinking water, surface water, groundwater, sediments, soils, hazardous waste sludges
Physical Properties	Surface water, groundwater
Cyanide	Groundwater, surface water, sediments, hazardous waste sludges
Microbiology	Drinking water, groundwater, Surface water

Fig. 2.6

EXAMPLE OF FIELD EQUIPMENT TABLE

Section 3.0
12-17-94
Page 3 of 5

Sampling Equipment Used by ORS Consultants

<u>Equipment</u>	<u>Construction</u>	<u>Use</u>	<u>Parameter Groups</u>
<u>Groundwater</u>			
Submersible Turbine Pump	SS Housing PVC tubing w/ check valve	Purging Sampling	Inorganic parameters Inorganic parameters
	SS Housing Teflon Tubing w/ check valve	Purging Sampling	All parameter groups Inorganic Parameters
Centrifugal Pump	Teflon Tubing w/ check valve	Purging Sampling	All parameter groups Inorganic Parameters
Baller	Stainless Steel	Purging Sampling	All parameter groups All parameter groups
<u>Surface Water</u>			
Sample Container	Glass Plastic	Sampling Sampling	All parameter groups Inorganic parameters
DO Dunker	Glass	Sampling	All parameter groups
<u>Miscellaneous Equipment</u>			
pH Meter	N/A	In-situ measurements	
Analyte-free water container	Glass Plastic	Transporting organic-free water Transporting analyte-free water	All parameter groups Inorganic parameters only
Gloves	Latex	Protection and Safety	All parameter groups

- a. Gas Chromatograph (detector types must be listed;
 - b. Mass spectrometer (identify whether MS or ITD);
 - c. ICP (specify whether sequential or simultaneous);
 - d. HPLC (detector types must be listed;
 - e. Types of spectrophotometers (UV-VIS, IR, etc.);
and
 - f. Atomic absorption spectrophotometers including
all auxiliary atomic absorption equipment (furnace,
flame, hydride, cold vapor, etc.)
3. Support Equipment should include:
- a. Analytical balances;
 - b. Microscopes;
 - c. TCLP and/or EP Toxicity equipment; and
 - d. Ovens, refrigerators, water baths and incubators.

2.3.3.7 Submission of CompQAP on Diskette

- a. All information listed above may be submitted on
diskette as an ASCII flat file. The specific format may be
obtained by writing or calling the QA Section or by
accessing the information through the QA Bulletin Board.
- b. A CompQAP that is submitted in this manner must be
accompanied by a signed and notarized hard copy of the
Statement of Intent.
- c. The initial screening review and determination of the
QA Plan status will be performed when the information is
uploaded into the QA data base. Additionally, the approved
capabilities of your organization will be immediately
accessible to DER Staff and the HRS Certification program.

2.4 COMPQAP FORMAT WHEN ADOPTING PORTIONS OF THE SOPS

The Comprehensive QA Plan must be submitted in the 15-element
format specified in the DER Manual for Preparing Quality
Assurance Plans (DER-QA-001/91, Revised 1992) and must contain
all required information for those sections or activities that
ARE NOT adopted by SOPs.

2.4.1 All topics specified by the DER Manual for Preparing
Quality Assurance Plans must be included in this document. The
subject matter may be by reference to adopted SOPs or must be
fully discussed in the QA Plan text.

2.4.2 A signed statement of intent (see Section 2.2 above) must
be submitted and will **REPLACE** the title page.

2.4.3 All activities or protocols from the SOP that are listed
on the statement of intent may be referred to by citation (see
instruction below). This means that the SOP information **DOES NOT**
NEED TO BE COPIED INTO TEXT OR ATTACHED AS AN APPENDIX.

2.4.3.1 A BLANKET REFERENCE TO THE ENTIRE SOP DOCUMENT IS NOT ACCEPTABLE. That is, you must specify the section or subsection that are pertinent to your operations.

2.4.3.2 References to the POTW Sludge Sampling Document or the EPA Region IV Standard Operating Procedures and Quality Assurance Manual must state the protocol and specify the section, page number(s) and revision date.

Example:

"Waste sampling in pits, ponds and lagoons will follow procedures outlined in the EPA Region IV SOP and QAM, Section 4.12.3, pp. 3-5, revised February 1991."

2.4.3.3 References to the SOPs written by the QA Section must state the protocol, the document title, section or subsection and revision date.

Examples:

"Sample Collection for Surface Water Samples will be performed in accordance with the documents and references specified in the DER Standard Operating Procedures for Laboratory Operations and Sample Collection Activities, Section 4.2.1, revised 6-92."

"Sample containers will be obtained by option b in the DER Standard Operating Procedures for Laboratory Operations and Sample Collection Activities, Section 4.4.1, revised 6-92."

"Laboratory sample documentation will be organized and maintained in accordance with the requirements specified in the DER Standard Operating Procedures for Laboratory Operations and Sample Collection Activities, Section 5.0, revised 6-92."

3.0 ORGANIZATION AND RESPONSIBILITIES

Every organization must identify a single individual who is ultimately responsible for the quality of the generated data. However, all individuals in the organization are responsible for assuring the accountability and reliability of the data. The following discussions assign certain tasks to various levels of responsibility. DER recognizes that the organization structure within a company may vary. With the exception of the QA Officer, the duties specified below may differ from suggested job titles and may be assigned to more than one person. However, DER expects that all tasks outlined below are performed by an individual or individuals within the organization.

3.1 QUALITY ASSURANCE OFFICER

The role of the Quality Assurance Officer (QAO) is one of oversight. Although the QAO usually coordinates and oversees data quality and corrective actions, the QAO must have the ability and authority to recommend and implement immediate corrective measures, without going through chains of command. Therefore, the organizational and functional position cannot be placed in direct lines of authority.

The Quality Assurance Officer must be able to objectively evaluate data and perform audits without outside influences. The responsibilities of the QAO may be divided among several individuals (i.e. corporate QAO, regional QA managers, etc.) and the designated QA Officer may be assigned other duties (project management, analytical work, etc.). However, these other responsibilities cannot bias the performance of these tasks:

1. Reviews quality control data to determine if test data is acceptable.
2. Updates or supervises update of all quality control markers: accuracy, precision and method detection limits.
3. Periodically performs systems audits to assure compliance with all quality assurance plans.
4. Oversees administration of performance audits.
5. Coordinates preparation of quality assurance reports to management, clients and regulatory agencies.
6. Coordinates and oversees preparation of quality assurance plans.
7. Reviews new or proposed protocols to determine appropriate use. Also reviews associated method validation information.
8. Reviews, in writing, initiated corrective actions to assure effectiveness. Recommends additional measures if necessary.

3.2 TECHNICIAN or ANALYST LEVEL

3.2.1 Sample Collection

1. Performs field measurement tests according to specified methodology.
2. Collects samples using approved techniques and appropriate equipment.
3. Assures that sample containers are properly and accurately labeled.
4. Assures that appropriate preservatives are added and that appropriate sample containers are used to collect required fractions.
5. Legibly documents all activities in field logs or field data sheets.
6. Assures that all identifying information is accurately recorded.
7. Identifies and/or documents potential quality control problems (ex. unacceptable calibrations, environmental conditions, etc.)
8. Maintains equipment in working condition, and documents all preventative maintenance and repairs.

3.2.2 Sample Analyses

1. Performs required analyses according to test methods specified by rule, permit or Quality Assurance Project Plans.
2. Assures that all analytical equipment has been properly calibrated before beginning tests.
3. Assures that all identifying information (including sample ID numbers) have been accurately transcribed into records or computer data bases.
4. Assures that all calculations are correct.
5. Assures that appropriate confirmatory tests or procedures have been completed.
6. Identifies, documents and begins corrective actions on any quality control problem which relates to the analytical test.
7. Maintains equipment in working condition, and documents all preventative maintenance and repairs.

3.3 SUPERVISORS and/or SUBSECTION/SECTION MANAGEMENT

1. Assures that all activities (either sampling or analysis) are performed according to methods and protocols specified in the QA Plan.
2. Reviews all analytical data by:
 - a. Checking documentation for completeness and proper sample identification.
 - b. Checking raw data for calculation, interpretation or clerical errors
 - c. Assuring that produced quality control data is acceptable

3. Coordinates analytical work or field activities to assure that completion of all tasks within established time frames.
4. Oversees preventative maintenance activities.
5. Evaluate and implement changes in methodology and quality control measures.
6. Identifies quality control problems and takes measures to correct or eliminate the problem source.
7. Assumes the responsibility for validating all data and assuring that final reports are accurate before final review by management.

3.4 PROJECT MANAGEMENT

1. Acts as a liaison between the client and the organization.
2. Oversees and coordinates project activities including workplans, quality assurance project plans and scheduling.
3. Reviews project data prior to final report to assure that all data (field and laboratory) are acceptable and within specified project objectives.

3.5 MANAGEMENT

1. Responsible for overall operation of the organization including fiscal resources and personnel.

4.0 SAMPLING PROCEDURES

4.0 GENERAL

4.0.1 Sample Collection Order

4.0.1.1 Samples shall be collected from the least to the most contaminated sampling locations within a site.

4.0.1.2 Unless field conditions justify other sampling regimens, samples shall be collected in the following order:

- a. Volatile Organic Contaminants (VOCs)
- b. Extractable Organics [includes Total Recoverable Petroleum Hydrocarbons (TRPH), Oil & Grease, Pesticides and Herbicides]
- c. Total Metals
- d. Dissolved Metals
- e. Microbiological
- f. Inorganics (Includes Nutrients, Demands and Physical Properties)
- g. Radionuclides

4.0.2 Use of Protective Gloves

4.0.2.1 Gloves serve a dual purpose: 1) protects the sample collector from potential exposure to sample constituents; and 2) minimizes accidental contamination of samples by the collector.

The Department recommends wearing protective gloves when conducting all sampling protocols, however, their use is not mandatory if:

- a. The sample source is considered to be non-hazardous; or
- b. The samples will not be analyzed for trace (i.e. part per billion level) constituents.

If worn, do not let gloves come into contact with the sample, the interior of the container or lip of the sample container.

4.0.2.2 Glove Types

The Department recommends the use of new, disposable, unpowdered latex gloves which should be changed and discarded after every sampling point. Other types of gloves may be used as long as the construction materials do not contaminate the sample or if internal safety protocols require greater protection.

Note that certain materials (as might be potentially present in concentrated effluent) may pass through certain glove types and be absorbed in the skin. There are permeability tables for differing types of gloves that might be advisable in certain situations.

4.0.3 Container and Equipment Rinsing

When collecting aqueous samples the sample collection equipment and non-preserved containers shall be rinsed with sample water before the actual sample is taken.

This protocol shall not be followed for:

1. Oil & Grease or TRPH - Neither the equipment (if used) nor the container shall be rinsed;
2. Microbiological or VOCs - Sample containers shall not be rinsed; or
3. Sample containers with premeasured preservatives in the container.

4.0.4 Dedicated Equipment Storage

1. All dedicated equipment (except dedicated pump systems or dedicated drop pipes) must be stored in a non-contaminating environment.

2. If possible, equipment should be stored in an area that is located away from the sampling site. If equipment must be stored in monitor wells, the equipment shall be suspended above the formation water. In addition, the monitor well shall be securely sealed to prevent tampering between sampling events and the equipment shall be decontaminated prior to use following protocols outlined in Section 4.1.4.

4.0.5 Fuel-powered Equipment and Related Activities

1. All fuel-powered equipment activities must be placed away from and downwind of any site activities (e.g. purging, sampling, decontamination). If field conditions preclude such placement, (i.e. the wind is from the upstream direction in a boat), the sampling activities shall be conducted as far away as possible from the fuel source(s) and the field notes must describe the conditions.

2. If possible, fuel handling (i.e. filling vehicles and equipment) should be done prior to the sampling day. If such activities must be performed during sampling, the personnel must wear disposable gloves. All fuel dispensing activities and glove disposal shall occur downwind and well away from the sampling activities.

3. If sampling at active gas stations, sample collection activities should be stopped during fuel deliveries.

4.0.6 Sampling Procedure References

1. The DER Sampling SOP contains text descriptions or approved references for all applicable sample matrices.

2. All equipment construction configurations shall follow Table 4.1 and Figures 4.1 and 4.2.

3. All sampling references MUST be available in the field for reference and consultation.

TABLE 4.1
SAMPLING EQUIPMENT - CONSTRUCTION AND APPROPRIATE USE

EQUIPMENT TYPE	CONSTRUCTION	USE	PERMISSIBLE PARAMETER GROUPS	RESTRICTIONS AND PRECAUTIONS (1)
WATER SAMPLING				
Groundwater				
Pumps: General Note: Pumps may not be used in sampling for volatile organic components except when pumps are permanently installed as a part of a drinking water system, or if positive displacement stainless steel and teflon bladder pumps are used. If installed as a part of a drinking water system, the material construction of the pump and holding tank must be noted in the field documentation.				
1. Positive Displacement Pumps				
a. Submersible (2) (turbine, helical rotor, gear driven)	<u>Housing (3)</u>	<u>Tubing (3)</u>		
	SS, Teflon	SS, Teflon	Purging	All parameter groups a,b; in-line check valve required
			Sampling	All parameter groups (excluding VOCs) a,b; in-line check valve required
	SS, Teflon	Non-inert(4)	Purging	All parameter groups a,b; in-line check valve required; polishing required (5)
			Sampling	Demands, Nutrients Metals, Radiochemistry none tubing non-metallic if not SS

FOOTNOTES

1. Restrictions/precautions listed on the last page of this chart, page 8.
2. Submersible pumps may be used for purging or sampling only if no other pumping device is available, since lines (power cords, gas pressure tubing) may not be (practically) constructed of inert materials.
3. This category refers to tubing and pump housings/internal parts that are in contact with purged or sampled water.
4. "Non-inert" pertains to materials which are reactive (adsorb, absorb, etc.) to the analytes being sampled. Materials include: polyethylene, PVC, and other plastics if organic are of interest and metallic equipment (brass, galvanized, and carbon steel, etc.) if trace metals are of interest.
5. "Polishing": When purging for organics, the entire length of tubing or portion which comes in contact with the formation water should be constructed of teflon or stainless steel. If other materials (i.e. PVC, HDPE, or polypropylene) are used, the following protocols must be followed: 1) contact with formation waters is minimized by slowly withdrawing the pump from the water column during the last phase of purging, thus removing from the well any water which may have contacted the exterior of the pump and/or tubing; 2) a single well volume must be removed with the sampling device before sampling begins. Tygon must never be used for purging when organics are of interest. NOTE: THE USE OF NON-INERT MATERIAL IS NOT RECOMMENDED.

TABLE 4.1, continued
SAMPLING EQUIPMENT - CONSTRUCTION AND APPROPRIATE USE

EQUIPMENT TYPE	CONSTRUCTION		USE	PERMISSIBLE PARAMETER GROUPS	RESTRICTIONS AND PRECAUTIONS
WATER SAMPLING					
Groundwater					
Pumps, cont.					
1. Positive Displacement Pumps, cont.					
a. Submersible, cont.					
	<u>Housing</u>	<u>Tubing</u>			
	Non-inert	Non-inert	Purging	All parameter groups	a,b; in-line check valve required; polishing required
			Sampling	Demands, Nutrients Metals, Radiochemistry	none tubing non-metallic if not SS
b. Bladder Pump (no gas contact)	SS, Teflon	SS, Teflon	Purging	All parameter groups	a,b
			Sampling	All parameter groups	a,b; bladder must be Teflon if sampling for organics
	SS, Teflon	Non-inert	Purging	All parameter groups	a,b; polishing required; this configuration not recommended
			Sampling	Demands, Nutrients Metals, Radiochemistry	none tubing non-metallic if not SS
	Non-inert	Non-inert	Purging	All parameter groups	a,b; polishing required
			Sampling	Demands, Nutrients Metals, Radiochemistry	none housing and tubing non-metallic if not SS
2. Suction Lift Pumps					
a. Centrifugal	N/A	SS, Teflon	Purging	All parameter groups	b; foot-valve required

TABLE 4.1, continued
SAMPLING EQUIPMENT - CONSTRUCTION AND APPROPRIATE USE

EQUIPMENT TYPE	CONSTRUCTION		USE	PERMISSIBLE PARAMETER GROUPS	RESTRICTIONS AND PRECAUTIONS
WATER SAMPLING					
Groundwater					
Pumps, cont.					
	<u>Housing</u>	<u>Tubing</u>			
a. Centrifugal, continued	N/A	Non-inert	Purging	All parameter groups	b; foot-valve required; polishing required
b. Peristaltic	N/A	SS, Teflon	Purging	All parameter groups	b; foot-valve or continuous pumping required
			Sampling	Demands, Nutrients Metals, Radiochemistry	none b; medical grade silicone tubing in pump head
				Extractable organics	b; configured as specified in Fig. 4.1
	N/A	Non-inert	Purging	All parameter groups	b; polishing required
			Sampling	Demands, Nutrients Metals, Radiochemistry	none b; medical grade silicone tubing in pump head
c. Pitcher, Hand (above ground)	N/A	SS, Teflon	Purging	All parameter groups	b; must use foot-valve
		Non-inert	Purging	All parameter groups	b; must use foot-valve; polishing required
d. Pitcher, Hand (submersible) (e.g. B-K pump)	Non-inert	N/A	Purging	All parameter groups	a; polishing required
3. Bailer	SS, Teflon		Purging	All parameter groups	none; not recommended
			Sampling	All parameter groups	none

TABLE 4.1, continued
 SAMPLING EQUIPMENT - CONSTRUCTION AND APPROPRIATE USE

EQUIPMENT TYPE	CONSTRUCTION	USE	PERMISSIBLE PARAMETER GROUPS	RESTRICTIONS AND PRECAUTIONS
WATER SAMPLING				
Groundwater, cont.				
3. Bailer, cont.	Non-inert	Purging	Demands, Nutrients	none; not recommended
			Metals, Radiochemistry	must be nonmetallic if not SS; not recommended
		Sampling	Demands, Nutrients Metals, Radiochemistry	none must be nonmetallic if not SS
Surface Water				
1. Nansen, Kemmerer, Van Dorn or equivalent	SS, Teflon or Teflon Coated	Specific Depth Grab Sampling	All parameter groups	none
	Non-inert		Demands, Nutrients Metals, Radiochemistry	none must be nonmetallic if not SS
2. Bailers	SS, Teflon	Grab Samples	All parameter groups	none
	Non-inert		Demands, Nutrients Metals, Radiochemistry	none must be nonmetallic if not SS
3. DO Dunker	SS, Teflon or Glass	Water Column Composite Sampling	All parameter groups	none
	Non-inert		Demands, Nutrients Metals, Radiochemistry	none must be nonmetallic if not SS
Field Filtration Units	N/A	Dissolved constituents	Demands, Nutrients Metals in groundwater and static wastewater & surface water	must use a 0.45u filter See Fig. 4.2; no intermediate vessels; positive pressure HDPE & Teflon bailers acceptable

TABLE 4.1, continued
SAMPLING EQUIPMENT - CONSTRUCTION AND APPROPRIATE USE

EQUIPMENT TYPE	CONSTRUCTION	USE	PERMISSIBLE PARAMETER GROUPS	RESTRICTIONS AND PRECAUTIONS
WATER SAMPLING				
Field Filtration Units, cont.			Metals in moving surface water (ie. river/stream)	must use positive pressure device, but an intermediate vessel may be used
SOLID SAMPLING				
Sediments/Soils				
1. Core Barrel (or liner)	SS, Teflon, Glass, Teflon coated, Aluminum	Sampling	All parameter groups	c,d,e
	Non-inert		Demands, Nutrients Metals, Radiochemistry	none d
2. Trowel, scoop, spoon or spatula	SS, Teflon or Teflon coated	Sampling and Compositing	All parameter groups	VOC samples may not be taken from composite samples
	Non-inert		Demands, Nutrients Metals, Radiochemistry	none must be nonmetallic if not SS
3. Mixing Tray (Pan)	SS, Teflon, Glass, Teflon coated, or Aluminum	Compositing or homogenizing	All parameter groups (except VOCs)	e
	Non-inert		Demands, Nutrients Metals, Radiochemistry	none e; must be nonmetallic if not SS
4. Shovel, Hand auger, Bucket auger	SS	Sampling	All parameter groups	none
	Non-SS		Demands, Nutrients	none
5. Split spoon	SS or carbon steel w/ Teflon insert	Sampling	All parameter groups	c,d
6. Shelby tube	SS	Sampling	All parameter groups	c
	Carbon steel		All parameter groups	c,d; samples for VOC and Metals must be taken from the interior of the core sample

TABLE 4.1, continued
SAMPLING EQUIPMENT - CONSTRUCTION AND APPROPRIATE USE

KEY TO RESTRICTIONS/PRECAUTIONS

- a. If used as a non-dedicated system, pump must be completely disassembled, if practical, and cleaned between wells.
- b. Delivery tubing must be precleaned and precut at the base of operations or laboratory (see Section 4.1.7).
- c. If samples are sealed in the liner for transport to the laboratory, the sample for VOC analysis must be taken from the interior part of the core.
- d. Liners must be constructed of stainless steel or a suitable non-metallic material. If a metallic (carbon steel, aluminum) liner is used with the core barrel, the samples for metals shall be taken from the interior part of the core sample.
- e. Aluminum foil, trays, or liners may be used only if aluminum is not an analyte of interest.

ACRONYMS

N/A = not applicable

SS = stainless steel

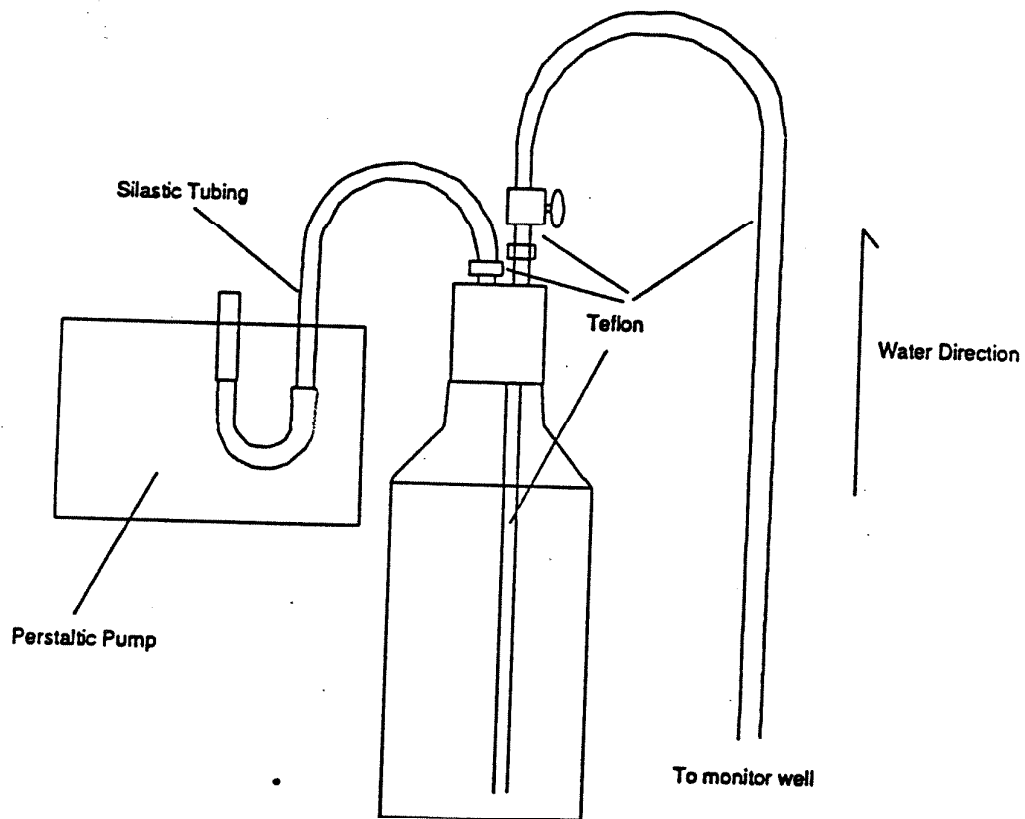
HDPE = high density polyethylene

PVC = polyvinyl chloride

VOC = volatile organic compound

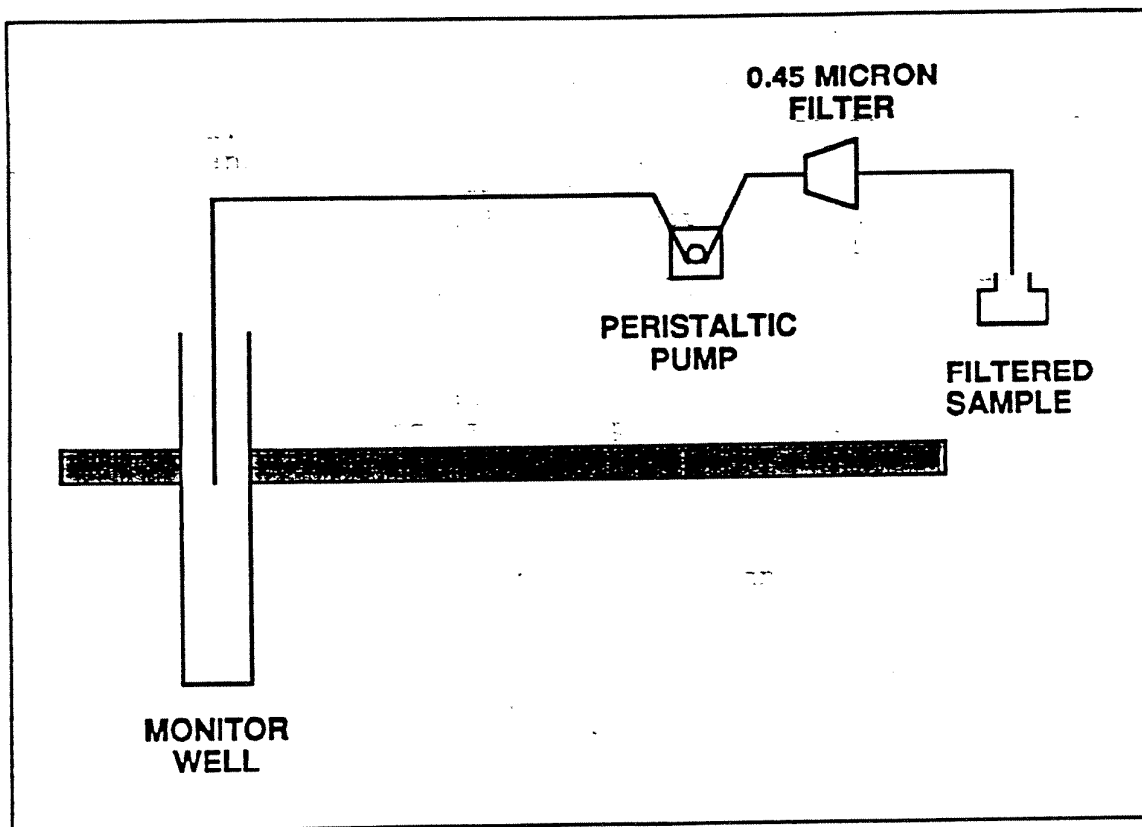
Fig. 4.1

**SYSTEM TO COLLECT SAMPLES FOR EXTRACTABLE ORGANICS
USING A PERISTALTIC PUMP**



The sample container is a glass bottle threaded to use either a permanent teflon-lined cap or a sampling cap with teflon insert similar to the design above.

Fig. 4.2
DIAGRAM OF RECOMMENDED FIELD FILTRATION SYSTEM FOR TRACE METALS



FIELD FILTRATION SCHEMATIC

1. Use one piece, molded, in-line high capacity disposable 0.45 micron filter.
2. Filter material should be non-contaminating synthetic fibers.
3. Filter should be placed on the positive pressure side of the peristaltic pump.
4. If well is deeper than 25 feet, a submersible pump or pressurized bailer may be necessary to bring the sample to the surface. The sample SHALL NOT be collected in an intermediate container.
5. At least one filtered equipment blank using deionized water must be collected and analyzed.

4.0.7 Preservation

1. All samples shall be preserved according to the requirements specified in Tables 4.2, 4.3, 4.4 and 4.5.
 - a. Based on directives from EPA, the holding times listed in the above-referenced tables SUPERSEDE any that might be discussed in individual analytical methods.
 - b. The holding times and preservation protocols specified by the tables listed above SHALL BE FOLLOWED.
2. The preservation protocols in the referenced tables specify immediate preservation. EPA has defined "immediate" as "within 15 minutes of sample collection". This definition shall be followed for all sample preservation. Therefore, DER recommends that the sampling team perform all preservation in the field.
3. 24-hour composite water samples are the exception to the "15-minute" criteria discussed above (see 4.2.4.6.b.5).

4.1 DECONTAMINATION

4.1.1 General

4.1.1.1 All equipment shall be cleaned in a controlled environment and transported to the field pre-cleaned and ready to use.

4.1.1.2 All equipment must be immediately rinsed with tap water after use, even if it is to be field cleaned for other sites.

4.1.1.3 Equipment that is only used once (i.e. not cleaned in the field) must be tagged with the sample location, returned to the in-house cleaning facility and cleaned in a controlled environment.

4.1.2 Reagents

4.1.2.1 Detergents specified in this document refer to Liquinox (or equivalent) or Alconox (or equivalent). See section 4.1.4.1 NOTE 3 for restrictions on use.

4.1.2.2 Solvents

a. The solvent used in routine cleaning procedures shall be pesticide grade or nanograde isopropanol. Pesticide grade and nanograde are synonymous.

1. Other solvents (i.e. acetone or methanol) may be used if they are pesticide grade. However, methanol is more toxic to the environment and acetone may be an analyte of interest for volatile organics.

2. Acetone SHALL NOT be used if volatile organics are of interest.

b. Precleaning heavily contaminated equipment (see section 4.1.4.1 NOTE 2.a) may be done with reagent grade acetone and hexane.

4.1.2.3 Analyte-free water sources shall be subject to the following criteria:

a. Analyte-free water is water in which all analytes of interest and all interferences are below method detection limits.

b. This type of water shall be obtained from a reputable source and documentation shall be maintained to demonstrate reliability and purity of analyte-free water sources (i.e. results from equipment blanks). As a general rule, the following types of water should be used:

1. Milli-Q (or equivalent) - suitable for all analyses
2. Organic-free - may be suitable for only VOCs and extractable organics
3. Deionized water - suitable for only inorganic analyses (metals, nutrients, etc.)
4. Distilled water - suitable for inorganics or microbiology

c. Analyte-free water shall always be used for blank preparation and for the final in-house decontamination rinse.

d. Analyte-free water shall be transported to the field in containers of suitable construction (see section 4.1.10).

4.1.2.4 Decontaminated field equipment shall be protected from environmental contamination by securely wrapping and sealing with one of the following:

- a. Aluminum foil - grocery store type is acceptable;
- b. Untreated butcher paper; or
- c. Clean, disposable plastic bags may be used if only inorganics are of concern OR the equipment is first wrapped in foil or butcher paper.

4.1.2.5 Acids

All acids used for cleaning shall be reagent grade or better:

a. 10% hydrochloric acid is prepared by mixing one part concentrated hydrochloric acid with 3 parts deionized water.

b. 10% nitric acid is prepared by mixing one part concentrated nitric acid with 5 parts deionized water.

Prepare acid solutions by slowly adding the concentrated acid to water.

4.1.3 Decontamination/Cleaning Protocols - General Considerations

4.1.3.1 All sampling equipment (bailers, lanyards, split spoons, etc.) that come in contact with the sample must be cleaned/decontaminated before use. The procedures that are applicable to the majority of sampling equipment are listed in Section 4.1.4.1. Protocols for other specialized equipment are outlined in Sections 4.1.4.2 through 4.1.11. Unless justified through documentation (i.e. company-written protocols and analytical records) and historic data (i.e. absence of analytes of interest in equipment blanks), the protocols outlined in Sections 4.1.4 through 4.1.11 shall be followed WITHOUT modifications.

4.1.3.2 Sufficient clean equipment should be transported to the field so that an entire study can be conducted without the need for field cleaning.

4.1.3.3 Unless otherwise justified, all field sampling equipment shall be precleaned in-house (office, lab, or base of field operations) prior to arrival on-site.

4.1.3.4 All cleaning shall be documented for each piece of field equipment.

- a. In-field decontamination shall be documented in the field records. These records shall specify the type of equipment that is cleaned and the specific protocols that are used (reference to internal SOPs for the cleaning protocols is acceptable).
- b. In-house cleaning records must identify the type of equipment (i.e. teflon bailers, PVC pump tubing, etc.), the date it was cleaned, the protocol or SOP that was used and the person who cleaned the equipment.

4.1.4 Decontamination/Cleaning Protocols - Sampling Equipment

4.1.4.1 General Cleaning Procedure for Teflon and Stainless Steel Sampling Equipment

This procedure shall be used when sampling for ALL parameter groups: extractable organics, metals, nutrients, etc.) or if a single decontamination protocol is desired to clean all Teflon and stainless steel equipment.

The cleaning procedures described below are for IN-FIELD cleaning, in-house cleaning must follow NOTE 1 of this section.

- a. Clean with tap water and lab grade soap (Liquinox or equivalent) using a brush, if necessary, to remove particulate matter or surface film (see NOTES 1, 2 and 3 of this section).
- b. Rinse thoroughly with tap water.
- c. If trace metals are to be sampled rinse with 10-15% reagent grade nitric acid (HNO₃). The acid rinse should not be used on steel sampling equipment (bailers, augers, trowels, etc.). See NOTE 4.
- d. Rinse thoroughly with deionized water (DI). Enough water shall be used to ensure that all equipment surfaces are flushed with water.
- e. Rinse twice with isopropanol. One rinse may be used AS LONG AS all equipment surfaces are thoroughly wetted with free-flowing solvent.
- f. Rinse thoroughly with analyte-free (see 4.1.2.3) water and allow to air dry as long as possible.

- g. Clean sampling equipment shall be wrapped (if appropriate) in aluminum foil, or in untreated butcher paper to prevent contamination during storage or transport to the field.
- h. If no further sampling is to be performed, equipment must be rinsed with tap water immediately after use.

NOTES:

1. In house Protocols require the following:
 - a. Protocols must include the use of HOT tap water and cleaning in a contaminant-free environment.
 - b. Analyte-free water must be used as a final rinse.
2. The DER QA Section does not recommend cleaning heavily contaminated equipment in the field. Such rigorous cleaning procedures should be performed at the base of operations. Cleaning at the base of operations or in the field require the following:
 - a. Prerinse equipment using the following solvents in the order described: acetone-hexane-acetone. The solvent rinse(s) must precede the soap and water wash described in the first step (a. above).
 - b. In extreme cases, it may be necessary to steam clean the field equipment before proceeding with step a. above.
 - c. If the field equipment cannot be cleaned utilizing these procedures, it should be discarded, unless further cleaning with stronger solvents and/or oxidizing solutions are effective.
3. Liquinox (or equivalent) is recommended by EPA, although Alconox (or equivalent) may be substituted if nutrients are not sampled.
4. If sampling for nutrients, a 10-15% reagent grade hydrochloric acid (HCl) rinse should be used (except stainless steel equipment). If BOTH metals and nutrients are to be sampled, the HCl rinse must replace the HNO₃ rinse, or the HNO₃ rinse must be followed by the HCl rinse.
5. Hot detergent solutions and water rinses are not required for in-field decontamination.

4.1.4.2 Teflon and glass equipment used to collect aqueous and solid samples for trace organics and metals [including oil & grease, TRPH, and total organic halogens (TOX)].

In-house cleaning - follow 4.1.4.1, see NOTES 1 and 2

In-field cleaning - follow 4.1.4.1, see NOTES 2 and 5

4.1.4.3 Stainless Steel or Other Metallic Equipment used to collect Trace Organics and Metals (includes oil and grease, TRPH, TOX)

In-house cleaning - follow 4.1.4.1, see NOTES 1 and 2, delete acid rinse

In-field cleaning - follow 4.1.4.1, see NOTES 2 and 5, delete acid rinse

4.1.4.4 Plastic equipment Used to Collect ONLY Trace Metals

In-house cleaning - follow 4.1.4.1 see NOTE 1, delete solvent rinse

In-field cleaning - follow 4.1.4.1 see NOTE 5, delete solvent rinse

4.1.4.5 All Equipment Used to Collect ONLY Nutrients (NH₄, NO₃, etc.) and Inorganic Anions (Cl⁻, SO₄, F⁻, etc.)

In-house cleaning - follow 4.1.4.1 see NOTES 1, 3, and 4; delete solvent rinse

In-field cleaning - equipment may be rinsed with analyte-free water immediately after use, then rinsed several times with sample water from the next sample.

4.1.4.6 All Equipment Used to Collect ONLY Demands and other inorganic non-metals

In-house cleaning - follow 4.1.4.1 see NOTE 1, delete solvent and acid rinses

In-field cleaning - equipment may be rinsed with analyte-free water immediately after use, then rinsed several times with sample water from the next sample.

4.1.5 Automatic Wastewater Samplers, Sampling Trains and Bottles

4.1.5.1 General - All ISCO and other automatic samplers shall be cleaned as follows:

a. The exterior and accessible interior (excluding the waterproof timing mechanisms) portions of automatic samplers shall be washed with laboratory detergent (see NOTE 3 above) and rinsed with tap water.

b. The face of the timing case mechanisms shall be cleaned with a clean, damp cloth.

c. All tubing (sample intake and pump tubing) should be checked and changed on a regular basis. Tubing shall be changed if it has become discolored (i.e. mold and algae) or if it has lost its elasticity.

d. New precleaned Silastic pump tubing (see 4.1.7.2 below) shall be installed.

4.1.5.2 ISCO (or equivalent) Automatic Sampler Rotary Funnel and Distributor

a. Clean with hot water, laboratory detergent (see NOTE 3 above) and a brush.

- b. Rinse thoroughly with deionized water.
- c. Replace in sampler.

4.1.5.3 ISCO (or equivalent) Sampler Metal Tube
Clean as outlined in 4.1.7.3 below.

4.1.5.4 All Automatic Sampler Headers

- a. Disassemble header and using a bottle brush, wash with hot water and phosphate-free laboratory detergent.
- b. Rinse thoroughly with deionized water.
- c. Reassemble header, let dry thoroughly and wrap with aluminum foil.

4.1.5.5 Reusable Glass Composite Sample Containers

- a. If containers are used to collect samples that contain oil, grease or other hard to remove materials, it may be necessary to rinse the container several times with reagent-grade acetone before the detergent wash. If material cannot be removed with acetone, the container must be discarded.
 - b. Wash containers thoroughly with hot tap water and laboratory detergent (see NOTE 3 above).
 - c. Rinse containers thoroughly with hot tap water.
 - d. If metals are to be samples, rinse containers with at least 10 percent nitric acid (see NOTE 4 above).
 - e. Rinse containers thoroughly with tap water.
 - f. Rinse containers thoroughly with deionized water.
 - g. If organics are to be sampled, rinse twice with solvent (isopropanol recommended) and allow to air dry for at least 24 hours.
 - h. Cap with aluminum foil, Teflon film or the decontaminated teflon-lined lid.
 - i. After using, rinse with tap water in the field, seal with aluminum foil to keep the interior of the container wet, and return to the laboratory or base of operations.
 - j. Containers should not be recycled or reused if:
 - 1. Containers used to collect in-process (i.e. untreated or partially treated) wastewater samples at industrial facilities;
 - 2. Any bottle having a visible film, scale or discoloration that remains after the above protocols have been used; or
 - 3. Containers were used to collect samples at pesticide, herbicide or other chemical manufacturing facilities that produce toxic or noxious compounds. Such containers shall be properly disposed of (preferably at the facility) at the conclusion of the sampling activities.
- If the containers described above are reused, no less than 10% of the cleaned containers must be checked for the

analytes of interest BEFORE use. If found to be contaminated (i.e. constituents of interest are found at MDL levels or higher), the containers SHALL BE DISCARDED.

4.1.5.6 Plastic Reusable Composite Sample Containers

- a. Use the protocols outlined in 4.1.5.5 above but omit the solvent rinse.
- b. Restrictions on reused or recycled containers listed in 4.1.5.5.j above shall be followed.

4.1.5.7 ISCO (or equivalent) Glass Sequential Sample Bottles for Automatic Sampler Based for Sequential Mode

Glass sequential sample bottles to be used in collecting inorganic samples shall be cleaned using the following protocol:

- a. Rinse with 10 percent nitric acid.
- b. Rinse thoroughly with tap water.
- c. Wash in dishwasher at wash cycle, using laboratory detergent cycle, followed by tap and deionized rinse cycles. Note: containers may be manually washed using protocol followed in 4.1.5.5 above. Solvent rinse should be deleted.
- d. Replace bottles in covered, automatic sampler base; cover with aluminum foil for storage.
- e. Rinse bottles in the field with tap water as soon as possible after sampling event.

4.1.5.8 ISCO (or equivalent) Glass Sequential Samples Bottles (Automatic Sampler Base for Sequential Mode) to be used for Collecting Samples for Organic Compounds

- a. Use cleaning protocols outlined in 4.1.5.5 above or use 4.1.5.7 above adding a solvent rinse after 7.c. Allow containers to air dry for at least 24 hours before use.
- b. Replace in covered automatic sampler base; and cover with aluminum foil for storage.

4.1.5.9 Bottle Siphons Used to Transfer Samples from Composite Containers

- a. Rinse tubing with solvent and dry overnight in a drying oven.
- b. Cap ends with aluminum foil and/or teflon film for storage.
- c. Seal in plastic for storage and transport.
- d. Flush siphon thoroughly with sample before use.

4.1.5.10 Reusable Teflon Composite Mixer Rods

- a. Follow protocols outlined in Section 4.1.4.1 above.
- b. Wrap in aluminum foil for storage.

4.1.6 Filtration Equipment

4.1.6.1 Dissolved Constituents using In-Line, Molded and Disposable Filter Units

- a. Peristaltic pump
 - 1. The peristaltic pump is cleaned per section 4.1.8.2.b "Pumps used for sampling"
- 2. The silastic pump tubing is cleaned per section 4.1.7.2
- 3. If Teflon tubing is used, it must be cleaned per 4.1.7.1
- 4. Other tubing types [high density polyethylene (HDPE), etc.] must be cleaned according to the appropriate protocol listed in 4.1.7
- b. Other equipment types (e.g. pressurized teflon bailer)
 - 1. Other types of equipment that utilize in-line, molded and disposable filters shall follow the appropriate cleaning regimen specified in Sections 4.1.4.1 through 4.1.4.6.
- c. Note: filtration units for dissolved metals shall follow the configuration and requirements outlined in Figure 4.1 and Table 4.1.

4.1.6.2 Dissolved Constituents using Non-disposable Filtration Units (i.e. syringes, "tripod assembly", etc.)

- a. Proceed with steps 1 through 5 of section 4.1.4.1, assembling and applying pressure to the apparatus after each rinse step (water and acid) to drive rinse material through the porous filter holder in the bottom of the apparatus.
- b. Remove and clean any transfer tubing per appropriate cleaning protocols (see section 4.1.7).
- c. Assemble the unit and cap both the pressure inlet and sample discharge lines (or whole unit if a syringe) with aluminum foil to prevent contamination during storage.
 - 1. Note: if the unit is to be used to filter only inorganic constituents (i.e. metals, nutrients, etc.), the unit may be sealed in a plastic bag to prevent contamination.

4.1.7 Sample Tubing Decontamination

4.1.7.1 Teflon Tubing

- a. New Tubing - if new tubing is used once and discarded, preclean as follows:
 - 1. rinse outside of tubing with pesticide-grade solvent
 - 2. flush inside of tubing with pesticide-grade solvent
 - 3. dry overnight in drying oven or equivalent (zero air, nitrogen, etc.)

b. Reused Tubing - tubing shall be transported to the field in precut, precleaned sections. The description below was written for in-lab cleaning only, FIELD CLEANING IS NOT RECOMMENDED. In-house cleaning shall follow these steps:

1. Exterior of the tubing must be cleaned first by soaking the Teflon tubing in hot, soapy water in a stainless steel sink (or equivalent non-contaminating material). Use a brush to remove any particulates, if necessary.
2. Take a small bottle brush and clean the inside of the tubing ends where the barbs are to be inserted.
3. Rinse tubing exterior and ends liberally with tap water.
4. Rinse tubing surfaces and ends with nitric acid, tap water, isopropanol, and finally analyte-free water.
5. Place tubing on fresh aluminum foil. Connect all of the pre-cut lengths of Teflon hose with Teflon inserts or barbs.

6. Cleaning configuration:

- a. Cleaning reagents (soapy water, acid, isopropanol, etc.) shall be placed in an appropriately cleaned container (2-liter glass jar is recommended)
- b. Place one end of the teflon tubing into the cleaning solution.
- c. Attach the other end of the teflon tubing set to the INFLUENT end of the peristaltic pump.
- d. The effluent from the peristaltic pump may be recycled by connecting tubing from the effluent to the glass jar with the cleaning reagents.
- e. Recycling as described above may be done for all reagents listed in 7 below EXCEPT the final isopropanol rinse and the final analyte-free water rinse. Disconnect the tubing between the effluent end of the pump and the jar of cleaning reagents.
 1. Isopropanol should be containerized in a waste container for proper disposal;
 2. Analyte-free water may be discarded down the drain.

7. Using the above configuration:

- a. Pump copious amounts of hot, soapy water through the connected lengths.
- b. Follow with tap water, 10% nitric acid, tap water, then isopropanol, and finally analyte-free water.
- c. During the nitric acid and solvent rinses, turn the pump off and allow the reagents to remain in the tubing for 15 minutes, then continue with the next

step. Pumping a liter (each) of the nitric acid and solvent should be sufficient, depending on the inside diameter of the hose.

d. Leave the Teflon inserts or barbs between the pre-cut lengths and cap or connect the remaining ends.

8. After the interior has been cleaned as described above, the exterior shall be rinsed with analyte-free water.

9. The connected lengths should then be wrapped in aluminum foil or untreated butcher paper and stored in a clean, dry area until use. Documentation for this cleaning shall be noted in the organization cleaning records.

4.1.7.2 Silastic Rubber Pump Tubing used in Automatic Samplers and other peristaltic pumps - this tubing need not be replaced if the sample does not contact the tubing or if the pump is used for only purging (i.e., not being used to collect samples). Tubing must be changed on a regular basis if used for sampling:

- a. Flush tubing with hot tap water and lab-grade detergent solution
- b. Rinse thoroughly with hot tap water
- c. Rinse thoroughly with DI water
- d. If used to collected metals samples, the tubing shall be flushed with 1+5 nitric acid, followed by thorough rinsing with DI water
- d. Install tubing in peristaltic pump or automatic WW sampler
- e. Cap both ends with aluminum foil or equivalent

NOTE: Tubing must be changed at specified frequencies as part the the preventative maintenance on the equipment.

4.1.7.3 Stainless Steel Tubing

The exterior and interior of stainless steel tubing shall be cleaned as follows:

- a. lab-grade soap solution with a long, narrow bottle brush
- b. rinse with (hot) tap water
- c. rinse with DI water
- d. rinse twice with isopropanol (if organics are sampled)
- e. completely air dry or thoroughly rinse with analyte-free water

4.1.7.4 Glass Tubing

- a. Use new glass tubing
- b. Rinse with isopropanol (if organics are of concern)
- c. Air dry for at least 24 hours
- d. Wrap in aluminum foil or untreated butcher paper to prevent contamination during storage

e. Discard tubing after use

4.1.7.5 Miscellaneous Non-Inert Tubing Types (tygon, rubber, HDPE, PVC, etc.)

a. New Tubing

1. As a general rule, new tubing may be used without preliminary cleaning.

2. New tubing shall be protected from potential environmental contamination by wrapping in aluminum foil, sealing in plastic bags or in the original sealed packaging.

3. If new tubing is exposed to potential contamination, the exterior and interior shall be thoroughly rinsed with hot tap water followed by a thorough rinse with deionized water.

4. If new tubing is to be used to collect samples, the tubing shall be thoroughly rinsed with sample water (i.e. pump sample water through the tubing) before collecting samples.

b. Reused Tubing

1. Flush tubing with soapy solution of hot tap water and laboratory detergent.

2. Rinse exterior and interior thoroughly with hot tap water.

3. Rinse exterior and interior thoroughly with deionized water.

4. If used to collect metals samples, the tubing shall be flushed with 10% nitric acid, followed by thorough rinsing with DI water

5. Wrap tubing and cap ends in aluminum foil and seal in plastic to prevent contamination during storage and transport.

4.1.8 Pumps

4.1.8.1 Submersible pumps

a. Pumps used for purging and sampling trace metals and/or organics

1. Construction of pump body and internal mechanisms (bladders, impellers, etc.), including seals and connections must follow Table 4.1

2. Choice of tubing material must follow Table 4.1

3. Pump exterior must be cleaned per section 4.1.4.1.

Note: the solvent rinse shall be deleted if the pump body is constructed of plastic (i.e. ABS, PVC, etc.)

4. Pump internal cavity and mechanism must be cleaned as follows:

- a. If for purging only, then the pump must be completely flushed with potable water prior to purging the next well.
- b. If for purging and sampling, then it must be completely disassembled (if so designed) and decontaminated between each well.
- c. If the pump cannot be (practically) disassembled, then the internal cavity/mechanism must be cleaned by pumping copious amounts of lab-grade soap solution, tap water, and DI water.
5. Teflon tubing will be cleaned per section 4.1.7.1.
6. Stainless steel tubing will be cleaned per section 4.1.7.3.
7. Cleaning of non-inert tubing shall follow the appropriate protocols in section 4.1.7 above (NOTE: very few options exist for non-inert tubing to be used for purging and/or sampling for trace organics).
- b. Pumps used for all other constituents (nutrient/inorganic anions)
 1. Pump construction - no restrictions
 2. Pump tubing material - no restrictions
 3. Scrub the exterior of the pump with appropriate metal-, phosphate- or ammonia-free detergent solution
 4. Rinse the exterior with tap water and deionized water
 5. Rinse interior of pump and tubing by pumping tap or deionized water through the system using clean bucket or drum.

4.1.8.2 Above Ground Pumps Used for Purging and Sampling

- a. Pumps used for purging only
 1. Exterior of the pump must be free of oil and grease
 2. Tubing choice must follow restrictions as specified in Table 4.1.
 3. Tubing coming in contact with formation water shall be cleaned according to the appropriate protocol for construction materials specified in section 4.1.7.
- b. Pumps used for sampling
 1. Exterior of pump must be cleaned with a detergent wash followed by tap and DI water rinses
 2. Tubing choice must follow restrictions as specified in Table 4.1.
 3. Tubing coming in contact with formation water shall be cleaned according to the appropriate protocol for construction materials specified in section 4.1.7.

4.1.9 Field Instruments and Drilling Equipment

4.1.9.1 Field Instruments (tapes, meters, etc.)

- a. wipe down equipment body, probes, and cables with lab-grade detergent solution,
- b. rinse thoroughly with tap water,
- c. rinse thoroughly with DI water, and
- d. wrap equipment in aluminum foil, untreated butcher paper or plastic bags to eliminate potential environmental contamination.

An optional isopropanol rinse may be performed if equipment comes in contact with contaminated water, etc.

4.1.9.2 Soil Boring Equipment

This pertains only to equipment that is NOT used to collect samples. Split spoons, bucket augers and other sampling devices must be cleaned per requirements listed in 4.1.4.1 or 4.1.4.3 above.

- a. The engine and power head, auger stems, bits and other associated equipment should be cleaned with a power washer, steam jenny or hand washed with a brush using detergent (no degreasers) to remove oil, grease, and hydraulic fluid from the exterior of the unit.
- b. Rinse thoroughly with tap water.

[[4.1.9.3 Well Casing Cleaning]]

These protocols are included and are meant as RECOMMENDATIONS for cleaning well casing and riser pipes. Recommendations from other DER programs, if different or more stringent, SHALL BE FOLLOWED.

[[a. The Department recommends only using casing that is designed for subsurface environmental groundwater monitoring. PVC pipe that is designed for well casing shall be transported to the field in original packing boxes.

b. Other PVC casing (for plumbing, etc. uses) shall be prepared for cleaning by sanding stencils (if present) on those portions of riser pipe that may come in contact with formation water. The ink used for stenciling may contribute to or contaminate the real samples. Casing that has been contaminated with grease, hydraulic fluid, petroleum fuel, etc. may require additional cleaning or deemed unusable.

c. All casing and riser pipe should be cleaned just prior to installation using the following protocol:

1. Steam clean all casing and riser pipe. Steam cleaning criteria shall meet the following: water pressure - 2500 psi, water temperature - 200F.
2. Rinse thoroughly with tap (potable) water. This tap water must be free of the analytes of interest, in effect analyte-free. Some potable water sources may have very low levels of contamination (e.g. benzene, trihalomethane, trichloroethane). A potable water source should only be used if it is known to be free of those contaminants that are being investigated.]]

4.1.10 Analyte-Free Water Containers

Analyte-free containers are defined by EPA to be glass containers. The DER QA Section will allow these containers to be made of glass, Teflon, polypropylene, or high density polyethylene (HDPE). It is strongly recommended that inert glass or Teflon be used for containerizing organic-free sources of water. Polypropylene is a good second choice. HDPE, though not recommended, is acceptable. Analyte-free water should not be left in these containers for extended periods, especially HDPE. These containers should be filled up for a single sampling event and then emptied at the end of the sampling day. EPA's cleaning procedure for glass (Teflon and polypropylene) is as follows:

4.1.10.1 New Containers

- a. Wash per instructions in 4.1.4.1 [(delete solvent rinse if plastic (HDPE or polypropylene) containers are being cleaned)].
- b. Cap with Teflon film, aluminum foil or the bottle cap.
Note: the bottle cap shall be equipped with a teflon liner. Aluminum foil or teflon film may be used as liner material.

4.1.10.2 Reused Containers

- a. Immediately after being emptied, cap with aluminum foil, teflon film or the container cap.
- b. Wash container exterior with lab-grade detergent solution and rinsed with DI water
- c. Rinse interior twice with isopropanol (delete if containers are plastic, see 4.1.10.1.a above)
- d. Rinse interior thoroughly with analyte-free water,
- e. Invert and allow to drain and dry
- f. Fill container with analyte-free water and cap tightly with aluminum foil, Teflon film or the container cap.
Note: the bottle cap shall be equipped with a teflon liner. Aluminum foil or teflon film may be used as liner material.
- g. Water shall not be stored for more than 3 days prior to a sampling trip.

4.1.11 Ice Chests and Shipping Containers

4.1.11.1 Wash exterior and interior of all ice chests with laboratory detergent (see NOTE 3).

4.1.11.2 Rinse with tap water and air dry before storage.

4.1.11.3 If the ice chest becomes severely contaminated with concentrated waste or other toxic or hazardous materials, it should be cleaned as thoroughly as possible, rendered unusable, and properly disposed.

4.2 AQUEOUS SAMPLING PROCEDURES

4.2.1 General

There are several requirements that are common to all types of surface water sampling events and are independent of technique. Several of these requirements are concerned with sample parameters that are inherently difficult to sample. In addition to the below procedures, overall care must be taken in regards to equipment handling, container handling/storage, decontamination, and record keeping.

4.2.1.1 Sample collection equipment and non-preserved sample containers must be rinsed with sample water before the actual sample is taken. Exceptions to this are: oil & grease, TRPH, microbiological, VOCs, or any pre-preserved container.

4.2.1.2 If protective gloves are used (see Section 4.0.2), they shall be clean, new and disposable. These should be changed prior to moving to the next sampling point.

4.2.1.3 Sample containers for source (i.e. concentrated wastes) samples or samples suspected of containing high concentrations of contaminants shall be placed in separate plastic bags immediately after collecting, preserving, tagging, etc.

4.2.1.4 If possible, ambient, or background samples should be collected by different field teams. If separate collection is not possible, the ambient or background samples shall be collected first and placed in separate ice chests or shipping containers. Highly contaminated samples shall never be placed in the same ice chest as environmental samples. It is a good practice to enclose highly contaminated samples in a plastic bag before placing them in ice chests. Ice chests or shipping containers with samples suspected of being highly contaminated shall be lined with new, clean, plastic bags.

4.2.1.5 If possible, one member of the field team should take all the notes, fill out tags, etc., while the other member does all of the sampling.

4.2.1.6 Teflon or glass is preferred for collecting samples where trace contaminants are of concern. Equipment constructed of rubber or plastic (e.g., PVC, Tygon, most Van Dorn Samplers) shall not be used to collect samples for trace organic compound analyses.

4.2.2 Special Parameter - Specific Handling Procedures

1. Since the concentration standards and/or guidance criteria for many analytes are in the (sub)parts per billion range, extreme care must be taken to prevent cross-contamination.

2. Most of the parameter groups listed in sections 4.2.2.1 through 4.2.2.8 below, shall be taken as grab samples unless Department requirements dictate otherwise. The exceptions are extractable organics and total metals which may be taken as composites, if required.

3. There is a greater chance of cross contamination when collecting composites because of increased sample handling and more equipment.

4. The following eight categories of parameters have specific sampling techniques and considerations which must be followed to collect unbiased, uncontaminated samples.

THE PROCEDURES OUTLINED BELOW SHALL BE USED FOR ALL AQUEOUS SAMPLING (I.E. SURFACE WATER, WASTEWATER, GROUNDWATER, STORMWATER ETC.).

4.2.2.1 Metals Sampling

a. Sample containers

1. New or properly cleaned plastic containers may be used for metals sampling. Glass bottles may also be used, but they are prone to breakage and occasionally react with the sample to either leach or adsorb metals from the glass itself.

2. All containers for metals sampling, new or previously used, shall be cleaned by following protocols outlined in Section 4.4.1.

3. Visually inspect polyethylene or glass containers for defects or contamination. Discard if defects are present or containers do not appear clean.

b. Preservation

1. Samples shall be preserved with nitric acid (HNO_3) of a grade that is suitable for use in trace metals analysis.

2. Preservation shall occur within 15 minutes of sample collection or filtration (if applicable) unless collected as a 24-hour composite (see 4.2.4.6.b.5).

3. Adequate HNO_3 shall be added per liter of sample to reduce the pH to below 2.0 to keep metals in solution and prevent them from adsorbing or absorbing to the container wall.

4. If only dissolved metals are to be measured, the sample shall be filtered immediately after sample

collection through a 0.45 um membrane filter. The sample shall not be preserved before filtration. See Table 4.1 for approved filtration equipment.

5. Samples submitted for Chromium VI should not be acidified.

c. Sample collection protocol:

1. Remove the cap from the sample container and rinse container with sample water (if not pre-preserved). Carefully pour sample into the container without allowing the sampling device to touch the rim of the sample container.

2. If adding preservatives in the field, the sample container should not be filled to capacity.

3. Acidify the sample to pH of 2 or less by adding a measured quantity of concentrated HNO₃ or 1+1 HNO₃ into the container.

4. NOTE: If containers are pre-preserved by a subcontract laboratory, the sample must be poured into the container slowly to prevent the acid from splattering. As a precautionary note, the addition of water to acid can generate enough heat to burn unprotected hands.

5. Tightly cap the sample container and shake to distribute the acid. Pour an aliquot of the acidified sample into a disposable container (e.g. sampling cup) or onto a piece of NARROW range pH paper to determine if the pH is less than 2.0. DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER!

a. Field experience has shown that UNDER NORMAL CIRCUMSTANCES, 2 ml of concentrated HNO₃ added to 250 ml of sample water will reduce the pH to less than 2.

b. If the pH is greater than 2, add additional MEASURED amounts of acid until the pH has been reduced.

b. Record the total amount of acid that was added to the sample. This documentation is necessary for the next site visit, since additional acid may need to be added to the sample on subsequent visits.

c. Acidify at least one of the equipment blank(s) with the GREATEST amount of acid that was required in the sample set and note the amount in field documentation.

6. Following proper sample preservation, tightly cap, affix a sample label, apply a seal (if required), and complete the COC or laboratory transmittal form.

7. Aqueous samples for metals need not be cooled to 4 C.

8. Make a note on the transmittal form identifying samples that have entrained sediment.

d. Filtration

1. For certain studies or projects, it may be necessary to obtain dissolved (i.e. filtered) samples. All samples that are filtered shall be identified in field notes and on final reports as "dissolved" or "filtered" metals.

2. Specific protocols for collecting dissolved metals from groundwater samples are discussed in Section 4.2.5.6.g. Filtered samples SHALL NOT be collected from groundwater sources unless:

a. The Department has required or approved the protocol; or

b. The organization feels that the contamination is directly related to the suspended material rather than the groundwater. In this case, BOTH unfiltered and filtered samples shall be collected and analyzed. The results shall be submitted to the Department for review. Based on the data, the Department may require continued collection of unfiltered samples, BOTH unfiltered and filtered samples or only filtered samples.

3. Surface water samples may use the sample protocols that are specified for groundwater (Section 4.2.5.6.g.) These protocols are recommended when sampling static surface water sources (i.e. subsurface samples from lakes, ponds, lagoons or ocean) since exposure to air can change the concentration of metals in solution. When sampling from moving sources (i.e. rivers or streams) or just below the surface, filtered samples may be collected into an intermediate container and filtered with syringe-type or tripod type filtration units.

4. Allowing a sample to settle and decanting the supernate (upper water layer) has been proposed as a means of removing suspended material. This technique MAY NOT be used for groundwater samples, and is not recommended for other sources because:

a. Settling times techniques are highly dependent on particle size and concentration and may not be reproducible;

b. Preservation for metals must occur within 15 minutes of sample collection which may not be sufficiently long for highly turbid samples to settle; and

b. The analytical results cannot be reported as "total" or "dissolved".

If this technique is used, the following protocols must be followed:

a. Samples shall not be acidified before settling occurs;

b. Total time for settling shall not exceed 15 minutes;

- c. The resultant supernate shall be carefully decanted into an appropriate container and preserved using protocols described above;
- d. Field notes shall specify the length of time the sample was allowed to settle, as well as observations on the initial and final (supernate); and
- e. The final report shall identify the technique that was used to collect the sample (i.e. decanted).
- f. NOTE: samples SHALL NOT be transported back to the laboratory for settling, UNLESS entire procedure (transport, settling, decanting and preservation) can occur within 15 minutes of sample collection.

4.2.2.2 Extractable Organics and Pesticides
Conventional sampling practices shall incorporate the following special considerations. Oil & Grease (O&G) and Total Recoverable Petroleum Hydrocarbons (TRPH) shall follow protocols outlined in Section 4.2.2.5 below.

a. Sample containers

- 1. Collect all samples in glass containers (1 liter to 1 gal.) with Teflon-lined caps. Note: Teflon containers are also acceptable.
- 2. Amber glass should be used for PAHs, nitrosamines, nitroaromatics, and isophorone.
- 3. Visually inspect glass bottles to assure that there are no glass or liner defects. If defects are present and/or the sample containers do not appear clean, the bottles must be discarded.
- 4. Sample containers must be cleaned according to the protocols specified in Section 4.4.1.
- 5. Composite samples from automatic WW samplers must be collected in refrigerated glass containers through Teflon tubing.

b. Preservation

- 1. Tables 4.2 and 4.3 must be followed to determine the specific preservation method for each group of organic compounds and pesticides.
- 2. All samples must be placed on wet ice immediately after collection. Samples must be maintained at a temperature of 4 C.
- 3. If the samples for pesticides cannot be extracted within 72 hours of collection, the sample pH must be in the range of pH 5 to 9. If needed, sample must be adjusted to the specified pH range with sodium hydroxide or sulfuric acid.
- 4. Other extractable samples need not be pH-adjusted with acid or base.

5. Samples must be extracted within 7 days of sample collection and the extracts analyzed within 40 days of extraction.

6. If residual chlorine is present, sodium thiosulfate must be added.

c. Sample collection protocol:

1. Sample bottles should be prerinsed with sample before collection, except Total Recoverable Petroleum Hydrocarbons (TRPH), Oil & Grease, etc. (see 4.0.3) or any prepreserved sample container.

2. Remove the cap from the bottle without touching the Teflon liner.

3. Do not allow the sampling equipment or hands to touch the rim of the sample container.

a. For bailer sampling, it may be necessary to utilize a stainless steel or Teflon delivery tube (fits into the bottom of the bailer).

4. Fill bottle with sample to almost full capacity.

5. Quickly place the Teflon lined cap over the bottle and tighten securely.

6. Affix a sample label, seal (if required), and complete the chain-of-custody form.

7. Put the sample bottle in a plastic sample bag and place on wet ice immediately.

8. Make a note on the lab transmittal form identifying samples that appear highly contaminated or exhibit other abnormal characteristics (i.e. foaming, odor, etc.).

4.2.2.3 Volatiles Sampling

a. Sample containers

1. Analysis of volatile organic substances requires a glass sample vial, sealed with a teflon-coated septum.

2. AT A MINIMUM, duplicate samples must be collected, although some laboratories require three or more vials. If the containers are not supplied by the laboratory, verify the laboratory's policy on how many vials are necessary and collect the specified number of vials.

3. Visually inspect the glass vials to assure that there are no glass or septum defects (e.g. rim must have not nicks or visible depressions); septum must not be deformed, etc.). If defects are present and/or sample containers or septums do not appear to be clean, the vials must be discarded.

4. Sample vials may be purchased precleaned from commercial vendors, or must be cleaned according to protocols outlined in Section 4.4.1.

5. NOTE: VIALS FOR VOCs ARE NOT RINSED WITH SAMPLE.

b. Preservation

1. Table 4.2 must be followed to determine the specific preservation method for each group of volatile organic compounds.

2. If residual chlorine is not present, the vials shall be filled with the sample, acidified (prepreserved containers are acceptable) with HCl and labeled "preserved".

3. If the volatile aromatics are to be analyzed within 7 days, HCl is not necessary.

4. Sodium thiosulfate must be added to samples with residual chlorine (see sampling protocols below).

5. Samples must be placed on wet ice immediately after sample collection. A temperature of 4 C must be maintained until the sample has arrived at the laboratory.

c. Sample collection protocols:

1. All fuel or exhaust sources which could cause VOC contamination must be situated well away and downwind of the sampling site (see Section 4.0.5).

a. If possible, fuels should be transported and stored in a separate vehicle from empty vials and collected samples.

b. All petroleum fueled engines (including the vehicle) must be situated downwind of the sampling operations.

2. Samples shall not be aerated during sample collection.

a. Extreme caution must be exercised when filling a vial to avoid any turbulence which could promote volatilization.

b. Carefully pour the sample down the SIDE of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a "convex meniscus."

3. Do not allow the sampling equipment to touch the rim of the sample container.

a. For bailer sampling, it may be necessary utilize a stainless steel or Teflon delivery tube or "pigtail" to obtain a gentle trickle of sample into the vial.

b. It is sometimes difficult to completely fill the vial directly from some waste streams. The sample may be collected in a precleaned intermediate sample collection device made of the appropriate materials (see Table 4.1) and carefully poured into the VOC vials.

4. The investigator must determine if the water to be sampled contains residual chlorine.

- a. If residual chlorine is present; add 10 mg of sodium thiosulfate to the vial (laboratory may supply vials with premeasured quantities).
 - b. Fill the vial 90% with sample.
 - c. Add four drops of concentrated HCl (more acid may be needed if the sample is known to contain high levels of bicarbonate or is otherwise buffered). Add additional sample (if needed) to create a convex meniscus and cap with zero headspace (see 5 below).
 - d. Label vial appropriately (preserved/sodium thiosulfate/acid).
5. The sample must be collected so that there are no air bubbles in the container after the screw cap and septum seal are applied.
- a. Vial must be filled so that the sample surface is above the container rim (convex meniscus).
 - b. The cap with the septum is then quickly applied (make sure teflon side of septum is down). Some sample may overflow, but air space in the bottle must be eliminated.
 - c. If acid has been added to the sample, tip the vial gently two or three times to distribute the preservative.
 - d. Turn the bottle over and tap it to check for bubbles.
1. If any are present, remove the cap, add a few more drops of sample, recap and test for bubbles.
REPEAT NO MORE THAN 3 TIMES.
6. Sampling and preservation containers may be prelabeled prior to any field activities. This may reduce confusion during a sampling event.
7. All the vials must be labeled. Make note in the field records of any samples that appear highly contaminated or appear to effervesce when acid is added.
NOTE: If the sample reacts with the acid by generating gas, DER recommends collecting unpreserved samples for analysis (seven-day holding time must be met).
8. Wrap each vial in bubble-wrap, or equivalent, and place each vial in a small ziplock-type bag and immediately place on wet ice.
9. Complete field records.
10. Protect samples from environmental contamination during storage and transport to the laboratory (4.2.2.3.c.1 above).
- a. As an added measure, replicate samples may be sealed in a container with vermiculite. This will add further protection from potential contamination.

4.2.2.4 Bacteriological Sampling

a. Sample containers

1. Samples must be collected in containers that have been sterilized according to Standard Methods (17th Edition) or the EPA's Microbiological Methods for Monitoring the Environment, 14th edition.

a. Presterilized Whirlpak bags (or equivalent) are typically used for sampling.

b. If Whirlpaks are not used, a 125 ml or larger sample container must be used to provide a minimum sample volume of 100 ml and adequate mixing space.

2. Unlined caps or ground glass tops shall be used to ensure complete sterilization of the container's closure.

3. Bottles and caps shall be sterilized according to protocols outlined in Section 4.4.1 or purchased presterilized from a commercial vendor.

b. Preservation

1. Samples shall be preserved according to Tables 4.2 and 4.5.

2. All samples shall be placed on wet ice immediately after sample collection.

3. When sampling water containing residual chlorine, a dechlorinating agent such as sodium thiosulfate must be added to the sample container.

a. The final concentration of sodium thiosulfate in the sample shall be approximately 100 milligram per liter (mg/L) in the sample.

b. As a general rule, this concentration may be achieved by adding 0.1 ml of a 10 percent solution of sodium thiosulfate to a 125 ml sample bottle.

c. The dechlorinating agent neutralizes any residual chlorine and will prevent further reaction between bacteria and chlorine.

4. ANALYSIS MUST COMMENCE WITHIN 6 HOURS FOR NON-POTABLE SOURCES AND 30 HOURS FOR POTABLE SOURCES. Special laboratory arrangements may need to be made so that the holding times are not compromised (may require local lab analysis).

c. Sample Collection Protocols:

1. Bacteriological sampling must always be collected as a grab sample and must never be composited.

2. The container must be kept unopened until the moment that the sample is collected.

3. DO NOT RINSE CONTAINER BEFORE COLLECTING SAMPLE.

4. When the Whirlpak bag or sample bottle must be lowered into the waste stream, either because of safety or

impracticality (manhole, slippery effluent area, etc.), care must be taken to avoid contamination.

5. Samples shall never be collected in an unsterilized sample container and transferred to a sterile container.

6. Be careful not to put fingers into the mouth of the container or on the interior of the cap.

7. If sampling intermediate sampling devices (i.e. bailers) or from in-place plumbing, the sampling device or the tap do not need to be disinfected (i.e. swabbing with alcohol or flaming with heat source).

a. Intermediate sampling devices shall be thoroughly rinsed with sample water prior to collecting the sample. For this reason, microbiological samples should be among the final samples that are collected from the sampling location.

b. Spigots shall be flushed at maximum velocity (see Sections 4.2.6 and 4.2.7) to purge the system and remove particulates. Sample flow shall be reduced to approximately 500 ml/min and allowed to run a few minutes before collecting samples (or microbiological samples may be collected last). **DO NOT STOP FLOW BEFORE OR DURING THE FILLING PROCESS.**

8. Surface water sample collection:

a. To sample with a rigid container, hold the bottle near the base and plunge neck downward, below the surface. Turn container until the neck points slightly upward with the mouth directed toward the current. Fill to within about 1/2 inch of the top and recap immediately.

b. To sample with a Whirlpak bag, open the bag by zipping off the top and pulling the white tabs to open the bag. Hold the bag in the hand or attach to a long handle and plunge neck downward and up in one sweeping arc.

9. Intermediate containers (i.e. bailers)

a. Obtain sufficient sample in the sample collection device to completely fill the sample container.

b. Begin pouring sample out of the device **BEFORE** collecting into the container.

c. Continue to pour sample out of the device, place container under flowing stream, and fill. **DO NOT STOP FLOW BEFORE OR DURING THE FILLING PROCESS.**

10. Wells with in-place plumbing, spigots and/or faucets

a. Samples shall be collected after flow has been reduced to 500/ml per minute.

b. Allow the water to flow at the reduced rate for a few minutes before collecting the sample, or collect all other samples prior to taking the microbiological sample.

c. DO NOT STOP FLOW BEFORE OR DURING THE FILLING PROCESS.

4.2.2.5 Oil and Grease (O&G) and Total Recoverable Petroleum Hydrocarbon (TRPH) Sampling

a. Sample Containers

1. Samples for O&G and TRPH shall be collected in 1 liter wide-mouth glass bottles.
 - a. The lid shall be teflon-lined.
 - b. If the cap is not teflon-lined, a sheet of teflon extending out from the lid may be used.
2. Visually inspect glass bottles to assure there are no glass or cap defects. If defects are present and/or sample containers do not appear to be clean, the bottles should be discarded.

b. Sample Preservation

1. Since losses of the product will occur on sampling equipment, composite samples shall not be collected.
2. The sample must be immediately preserved by adding H₂SO₄ or HCl to reduce the pH to 2.0 or less.
3. Samples must be placed on wet ice immediately after preservation. The temperature of the sample must be maintained at 4 C until received and processed by the laboratory.

c. Selection of Sampling points

1. Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. Since it is very difficult to collect a representative ambient sample for oil and grease analysis, the sampler must carefully evaluate the location of the sampling point.
2. The most desirable sampling location for both O&G and TRPH is the point where greatest mixing is occurring. Quiescent areas should be avoided, if possible.
3. Skimming the surface for the sample is unacceptable.
4. For compliance samples at a facility you may want to take samples at the worst place.
5. Neither the container, nor the sampling device, shall be rinsed before the actual sample is taken.
6. COMPOSITE SAMPLES SHALL NOT BE COLLECTED. If composite data is required, individual grab samples that are collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

d. Sampling Protocols

1. Sampling for these products is unique because they are immiscible and tend to adhere to the sampling device; therefore, these sample shall always be a grab sample.

2. The sample, when collected, should not be transferred to another container. The analytical methods require the use of the entire sample. In addition, the sample container must be rinsed with solvent as a part of the laboratory analytical process. Therefore these samples must be separate and discrete samples that will be used only for the O&G or TRPH analysis.

3. Remove the cap from the glass bottle without contacting the interior of the container or lid.

4. DO NOT RINSE THE BOTTLE WITH SAMPLE WATER.

5. Whenever possible samples should be collected directly into an unpreserved sample container. If intermediate sampling equipment is used, do not allow the sampling equipment to touch the rim of the sample container. AUTOMATIC SAMPLERS SHALL NOT BE USED TO COLLECT THESE TYPES OF SAMPLES.

6. Fill the bottle with the sample water to almost full capacity.

7. Add preservatives and check the pH using the protocols outlined in 4.4.2.2.b.

8. Quickly cap the container and tighten securely.

9. Affix a sample label, seal (if required), and complete the chain-of-custody form.

10. Protect glass container from breakage ("bubble wrap" is recommended), place the sample bottle in a plastic sample bag and keep it cool to 4 C on wet ice.

11. Make a note on the lab transmittal form identifying samples that may be highly contaminated or any other unusual observations.

4.2.2.6 Radiological Sampling (Excludes Radon)

a. Sample containers

1. Polyethylene, polyvinyl chloride (PVC), or Teflon containers are recommended for collecting radioactive samples because these containers are less adsorbent than glass or metal containers. Since radioactive elements are often present in extremely low quantities, a large fraction of the elements may be lost by adsorption on containers or glassware surfaces used in analyses. This loss may, in turn, cause a loss of radioactivity and possibly contaminate subsequent samples due to reuse of inadequately cleaned containers. Glass bottles are also more susceptible to breakage during handling than plastic containers.

2. Containers shall be cleaned according to the protocols specified in Section 4.4.1.

b. Preservation

1. The preservation technique for radiological sampling is acidification to a pH of less than 2.0 with concentrated or 1+1 nitric acid (HNO₃).

2. The pH shall be checked in the field following the protocols described 4.4.2.2.b.

c. Sampling Protocols

Prior to sampling, the area may be surveyed with a beta-gamma survey instrument, such as a Geiger-Mallor meter. If radiation levels are above instrument background, the investigator should consult a radiation safety specialist to determine appropriate safety procedures.

4.2.2.7 Radon Sampling

Radon is a gas and is easily removed from water sources. Therefore, the same precautions and care used to collect volatile organic samples shall be followed. It is extremely important to minimize contact with air during sample collection.

a. Sample Containers

1. Glass sample vials shall be obtained from the analyzing laboratory and shall contain a premeasured portion of the scintillation "cocktail".

2. A minimum of two samples is required. Laboratories are expected to provide the sampler with the requisite number of containers for each sample.

b. Preservation - the scintillation cocktail is the only required preservative.

c. Sampling Protocol

The laboratory should provide specific sample collection instructions that must be followed. These protocols should included proper handling as well a sample size and packing instructions. The following are general instructions that should be used:

1. Carefully fill a syringe (usually 10 ml) with sample water so that air bubbles are not pulled in with the sample before, during or after filling.

2. Place the tip of the syringe BELOW the scintillation cocktail and slowly dispense the sample BENEATH the cocktail surface.

3. Replace lid and cap tightly.

4. Generally the vial is used in the laboratory analytical instrument and labels or ID numbers on the sides

of the containers may interfere with the analysis. Check with the laboratory for proper placement of labels or field ID numbers.

5. Ship in an upright position in the shipping containers that have been provided by the laboratory. If none are provided, protect vials from breakage (bubble wrap is recommended), segregate replicate samples in separate plastic bags, and ship to laboratory in an upright position.

- 4.2.2.8 Cyanide Sampling s

Cyanide is a very reactive and unstable compound. Cyanide should be analyzed as soon as possible after collection. Note that the currently approved method (Standard Methods and EPA MCAWW) is being rewritten due to some inaccuracies in the method language.

a. Sample Containers -

1. The sample container shall be polyethylene or glass.
2. Containers shall be cleaned in accordance with protocols outlined in Section 4.4.1 of this manual.

b. Preservation

Sulfides tend to be a regional problem in Florida and can cause interference with cyanide analyses.

1. Proper preservation of aqueous cyanide samples must follow the standard procedures listed below.

2. All samples shall be tested for sulfides with test papers (EM, Chemometrics) or kits (HACH). However, these tests may not detect sulfides in low enough concentrations to be useful.

3. Ultimately, all samples shall be preserved to a pH of greater than 12 with sodium hydroxide and placed on wet ice immediately after preservation. A temperature of 4 C shall be maintained until analysis begins at the laboratory. The pH of the samples shall be checked to assure proper pH (see 4.4.2.2.b).

4. Samples that may or may not contain sulfides must be preserved in one of two ways: (1) samples are tested for sulfides, preserved with NaOH to a pH>12 and sent to the lab for analysis within 24 hours; or (2) tested for sulfides and pretreated as follows:

a. Test for sulfides

1) Samples with visible particulates must be filtered. Keep this filter (#1);

2) Sulfides may be tested with test papers (EM, Chemometrics) or kits (HACH). Tests may not detect sulfides in low enough concentrations to be useful;

3) Remove sulfide by adding cadmium (or lead) nitrate (or carbonate) powder to the sample (filtrate) to precipitate the sulfides;

4) Test for presence of sulfides. Repeat steps 2 and 3 until the test shows no sulfides are present;

5) Remove the precipitate (sulfides) from the sample by filtration and discard this filter.

b. Preservation

1) Reconstitute the sample by adding the solids collected on filter #1 back into the filtrate;

2) Add NaOH until the sample pH > 12 and cool to 4 C;

3) Maximum holding time is now 14 days;

4) Equipment blanks must be handled the same as real samples.

5. All samples known to contain oxidizing agents (chlorine) must first be tested as follows:

a. Test sample with KI-starch paper;

b. Add a few crystals of ascorbic acid, mix sample and retest.

c. Continue to add ascorbic acid until the test is negative;

d. Add an additional 0.6 grams of ascorbic acid per liter of sample to remove chlorine.

4.2.3 Surface Water Sampling

4.2.3.1 Introduction and Scope

[[Surface water samples may be taken for several reasons. Ambient conditions can be documented for a single point in time to determine if threshold Water Quality parameter limits are being met. Long-term sampling of a site or watershed may be used to document trend analysis. All of these items can be used to determine if a particular surface water body (or segment) is meeting its designated use. Sediment sampling may provide confirmatory information as to contaminants that are presently contained in the water column as well as an historical account of those contaminants that settled or precipitated out of the water column. Physical conditions of the water body and physico-chemical properties of the contaminants will determine their movement in and out of the water column. Additional information on data use, study objectives, etc. can be found in the EPA Region IV SOP & QAM, February 1991, Section 4.8.1. This document will henceforth be referred to as the EPA SOP.

Sample site selection is dependent upon the three major groupings; lakes, estuaries, and streams. Sites may have already been predetermined by a EPA, DER, WMD, etc. permit or by designation as a permanent monitoring station (PMS). If these

sites have not already been designated as above or by the DER water or waste program for which this sampling event has been required, then refer to the general descriptions in the EPA SOP, Sections 4.8.2. The EPA document provides generalized descriptions for proper site choices. Only sampling procedures will be described in the following sections.]]

This section presents the standard operating procedures that shall be employed during field investigations to ensure that representative surface water samples are collected. The particular surface water types that will be addressed include; static lakes, ponds, and impoundments; tidally-influenced estuarine areas; as well as streams and rivers. The importance of proper sampling of this varied matrix cannot be overemphasized. Proper sampling for the submerged (or emergent) sediments that underlie these surface water bodies is equally important. Care should be taken so that samples are neither altered nor contaminated by sample handling procedures.

This section discusses grab, depth-specific, and depth composited surface water samples. Information regarding flow- or time-weighted aqueous sampling is found in the Wastewater Sampling section.

4.2.3.2 General

Access will be left up to the sampling group. Ease of access should not be the main criteria for sampling site choice. If sampling from a bridge, by boat, or by wading, there are certain precautions that must be considered:

- a. If sampling with a boat, samples should be taken from the bow, away and upwind from any gasoline outboard engine (see 4.0.5.1).
- b. Collect samples upstream from the body when wading in to collect water samples.
- c. Care should be taken not to disturb sediments when taking samples in lakes, ponds, impoundments.
- d. If water samples and sediment samples are to be taken from the same area, the water samples must be taken first.
- e. Sampling at or near structures (dams, weirs, bridges) may not provide representative data because of unnatural flow patterns.
- f. Surface water and/or sediments should always be collected from downstream to upstream.

4.2.3.3 Sample Acquisition

Three (3) types of general sample acquisition methods will be discussed: grab samplers; mid-depth samplers; and composite samplers.

a. Grab Sampling

1. If the sample media is homogenous, grab samples are an effective and simple technique. If homogeneity is not known (and should never be assumed) then other techniques must be used.

2. Surface grabs using unpreserved sample containers are encouraged since the sample container is used for collecting the sample and, after appropriate preservation, the same container can be submitted for laboratory analysis. This reduces sample handling and eliminates potential contamination from other sources (i.e. additional sampling equipment, environment, etc.). If the laboratory provides prepreserved sample containers, the sample shall be collected in an UNPRESERVED sample container or with sampling equipment. The container or equipment shall be of appropriate construction (see Table 4.1) and the sample shall be transferred immediately into the prepreserved sample container

3. Simple Grab Samples - Typical sample collection equipment includes not only sample containers, but also precleaned beakers, buckets, and dippers. These samplers must be constructed appropriately (including handles):

a. Sample Container (unpreserved)

1. submerge the container, neck first into the water,

2. invert the bottle so the neck is upright and pointing into the water flow (if applicable),

3. return the filled container quickly to the surface,

4. shake to rinse the interior surface of the container and pour contents out downstream of sample location (see restrictions outlined in 4.0.3)

5. Collect sample as described in steps 1,2 and 3 above.

6. pour out a few mls of sample downstream of sample collection. This allows for addition of preservatives and sample expansion

7. Securely cap container, and label.

b. Intermediate vessel

1. Collect sample as outlined in 3.a above.

2. Pour into prepreserved sample container (or field preserve per Section 4.4.2.2.a), check pH per Section 4.4.2.2.b (if applicable), cap, and label.

4. Pond Sampler - Another effective technique is using a pole-mounted flask, beaker, or container. A long, telescoping pole (swimming pool supply) is outfitted with a (non-contaminating) clamp. An appropriately constructed and shaped container is fitted into the clamp. In this way the sample can be taken away from the shore, boat, bridge, etc. and at a specific spot. The sampling vessel can be

constructed of all-inert material so that all parameters can be sampled.

- a. Submerge the clamped container neck first, invert and withdraw from water.
 - b. Be careful not to entrain sediments or skim the water surface.
 - c. Rinse container (restrictions specified in Section 4.0.3 must be observed), resubmerge and collect sample. Retrieve the pole, clamp, and container and fill the sample containers.
5. Pump and Tubing - Although the use of a peristaltic pump and tubing can provide an adequate mid-depth or depth composite, it can also be used for taking a grab sample. This would be especially helpful if a large amount of sample is needed.
- a. Lower appropriately precleaned tubing to a depth just below the water surface (6 - 12 inches).
 - b. Turn the pump on.
 - c. Allow several tube volumes to pump through the system to acclimate the tubing.
 - d. Make sure the tubing does not come out of the water and inadvertently pull some surface skim water through the tubing (this may bias sample results).
 - e. Fill the individual sample bottles via the discharge tubing.

NOTE: THIS TECHNIQUE IS NOT ACCEPTABLE FOR OIL & GREASE, TRPH OR VOCs. It is not recommended for extractable organics (requires the organic trap setup, see Fig. 4.1) or microbiologicals (new, unused tubing, including tubing in the sampling head are required at each sampling location).

b. Mid-Depth Sampling

1. Mid-depth samples or samples taken at a specific depth can approximate the conditions throughout the entire water column.
 - a. One sample may be taken when the water body is assumed to be homogenous.
 - b. Additional samples can be taken from different depths at one spot to get a much more exact approximation of the conditions.
 - c. Many times a single site will be sampled from: just below the surface; mid-depth; and just above the bottom (sediment).
 - d. Accurate sampler location is imperative for this sampling technique.
2. The equipment that may be used for this type of sampling are: a device designed specifically for

depth-specific sampling (kemmerer, niskin, beta, etc.); pumps with tubing; or double check valve bailers.

a. Samplers are available from many manufacturers and in a variety of configurations and construction materials.

b. When purchasing and choosing a device for a particular sampling event, please be aware that certain construction material details may preclude its use for certain parameters (see Table 4.1):

1. Many kemmerer samplers are constructed of plastic and rubber which precludes their use for all organic sampling parameters (volatile and semivolatile).

2. Some of the newer devices are constructed of stainless steel or are all-Teflon or Teflon coated. These would be acceptable for all parameter groups without restriction.

3. NOTE THAT ALL RELATED COMPONENTS (STOPPERS, ETC.) MUST BE CONSTRUCTED OF INERT MATERIAL AS WELL IF ORGANICS ARE TO BE SAMPLED.

3. Kemmerer, niskin, and beta type devices

a. Separate and specific deployment discussions are not provided in this document. Manufacturers suggestions shall be followed for specific procedures.

1. Before lowering the sampler, measure the water column to determine maximum depth and sampling depth.

2. The line attached to the sampler should be marked with depth increments so that the sampling depth can be accurately recorded.

3. When dropping the sampler to the appropriate depth, it should be done slowly so that sediments are not stirred up.

4. Once the desired depth is reached, send the messenger weight down to trip the mechanism.

5. The sampler should be lowered and retrieved slowly.

6. The first sample shall be discarded into a bucket (to be dumped at conclusion of sampling).

4. Double check-valve bailers

a. Sampling with these type of bailers shall follow the same protocols outlined in 3 above.

b. Although not designed specifically for this kind of sampling, it will be acceptable when a mid-depth sample is required.

c. Note: this sampler does not perform as well as the devices described above or the pump and tubing described in the next section.

d. As the bailer is dropped through the water column, water will be displaced through the body of

the bailer. The degree of displacement is dependent upon the check valve ball getting out of the way and allowing water to flow freely through the bailer body.

e. An open-top bailer may also be used, but is not recommended. The open-top arrangement will not prevent water from being exchanged in the top portion of the bailer.

f. A closed-top bailer does not allow free water displacement on descent at all and is not acceptable.

g. The bailer should be dropped slowly to the appropriate depth. Upon retrieval, the (two) check valves seat, preventing water from escaping out of or entering the bailer.

5. Pump and Tubing

a. The most portable pump for this technique is a (12 volt) peristaltic pump.

b. Appropriately precleaned silastic is required in the pump head and HDPE, Tygon, etc. tubing is attached to the pump.

c. Measure the water column to determine the maximum depth and the sampling depth.

d. Tubing will need to be tied to a stiff pole or be weighted down so the tubing placement will be secure.

1. A lead weight should not be used.

2. Any dense, non-contaminating, non-interfering material will work (brick, SS weight, etc.).

3. Tie the weight with a lanyard (braided or monofilament nylon, etc.) so that it is located below the inlet of the tubing.

e. Turn the pump on and allow several tubing volumes of water to be discharged before taking the first sample.

f. Sample containers are then filled in the proper order, preserved, labeled, and placed on ice (if required).

c. Composite Sampling

Composite sampling will be used when a single sample that approximates a given depth interval is desired. Any of the devices described in mid-depth sampling can be used for composite sampling. The devices must be activated or manipulated in a way that the actual volumes sampled within the interval are ALL EQUAL PROPORTIONS. For instance, because of head pressure, the pump and tubing will pull a greater volume of sample at 5 feet in comparison to 20 feet. For this reason, great care must be used so that sample results are not biased. The use of the niskin,

kemmerer, beta, bailers, etc. containers may take more time, but sample control will be greater.

4.2.4 Wastewater Sampling

4.2.4.1 Introduction and Scope

Prior to mobilizing, the sampler must decide what kind of samples to collect, for what parameters, and where to collect them. This section will provide the guidelines as to what kind of sample should be collected, where it should be collected, and how to collect it. It will also discuss choosing parameters for analysis. Care must be taken to ensure that the sampling location is correct and that the samples are representative of the discharge. The site must be consistent with its permit.

This section is also applicable to stormwater runoff sampling.

[[4.2.4.2 Site Selection

The following discussion deals with site selection, sampling points and sample collection strategies and are for educational and informational use.

a. Samples shall be collected at the appropriate permitted locations and at locations necessary to determine environmental impact (e.g., effluent outfall, ground water monitoring well, land application site, receiving water stations).

1. If the permitted sampling point is not adequate for collecting a representative sample, the sampler should determine the most representative sampling point available and collect samples at both locations. This should be done with the concurrence of DER and the facility.

2. The reason should be documented in the field log for later resolution if challenged and for consideration of correcting the sampling point during permit renewal.

3. Recommendations for a change in sampling location should be given to the DER permit writer. Sample locations should be specified in such detail that anyone could follow the directions to the site and collect a sample at the same place.

b. The following are the most common locations for collecting a sample at a facility. They may or may not be described in the permit.

1. Effluent - Effluent samples should be collected at the site specified in the permit, or if no site is specified in the permit, at the most representative site downstream from all entering wastewater streams prior to discharge to the appropriate disposal method (e.g., surface

water discharge, ground water discharge, wetlands discharge, deep-well injection).

2. Influent - Influent wastewaters are preferably sampled at points of highly turbulent flow in order to ensure good mixing; however, in many instances the most desirable location may not be accessible. Preferable influent wastewater sampling points include:

- a. the upflow siphon following a comminutor (in absence of grit chamber);
- b. the upflow distribution box following pumping from main plant wet well;
- c. aerated grit chamber;
- d. flume throat;
- e. pump wet well when the pump is operating.

In all cases, influent samples shall be collected upstream from recirculated plant supernatant and residuals, and the sample collected should be completely untreated.

3. Internal Outfalls - Internal outfalls are to be sampled as specified in the permit or consent order.

- a. This type of sampling occurs on special projects, on industries or domestic facilities undergoing a diagnostic inspection, on treatment trains in a facility which are essential to the final effluent quality, and on facilities which occupy watersheds (e.g., phosphate mines).

- b. Sampling of internal outfalls is uncommon, but can be useful in identifying potential equipment problems and unidentified contaminant sources.

4. Groundwater Monitoring Wells - Ground water monitoring wells should be sampled as described in the Groundwater Section.

5. Groundwater Discharge Sites - Sample collection of discharges to land application sites and underground injection wells are best taken at the effluent sampling points.

6. Residuals - Residual samples should be taken in accordance with EPA's POTW Sludge Sampling and Analysis Guidance Document, 1989.

7. Residual Sites - Samples taken at a residuals disposal site are taken to determine build-up of pollutants in the soils.

8. Pond and Lagoon Sampling - Generally, composite wastewater samples should be collected from ponds and lagoons. Even if the ponds or lagoons have long retention times, composite sampling is necessary because of the tendency of ponds and lagoons to stratify. However, if dye studies or facility data indicate a homogeneous discharge, a grab sample may be taken as representative of the waste stream.

9. Surface Water Sites - The location of surface water sampling sites for wastewater facilities depend on whether the facility discharges to a unidirectional flowing body of water (e.g., stream, river) or a non-unidirectionally flowing body of water (e.g., tidally influenced coastal rivers, bays, estuaries, lakes).

a. Unidirectional Flow Streams - The upstream control site, if one exists, should be just far enough upstream to be out of the influence of any effluent. This may entail going farther upstream to avoid any potential groundwater contamination from adjacent spray irrigation sites or percolation ponds.

1. The downstream or test site should be at the edge of any mixing zone (if there is one) and far enough downstream to be in the peak zone of impact. These sites should be determined on a case-by-case basis.

2. The control and test sites should be matched as closely as possible on habitat structure (e.g., flora, pool/riffle type, shading) based on a habitat analysis.

3. If there is no upstream site (e.g., when the discharger currently forms the upstream flow), then a reference site should be chosen in a nearby stream based on a habitat analysis.

b. Nonunidirectional Flow Water Bodies - For tidally influenced rivers, bays, estuaries, swamps, lakes, ponds, and other water bodies which don't have unidirectional flow, an unimpacted reference site must be selected.

1. The site should be matched carefully on habitat structure (e.g., type, sediments, stream order/size, type of drainage) to ensure you are comparing the same type of sites.

2. The outfall should have two test sites either at the edge of the mixing zone or in the predicted zone of impact. The two sites should be located in different directions depending on where the effluent plume would be expected during tidal changes, in the direction of prevailing wind, or along anticipated flow gradients even though undetectable.

10. Effluent limits in a permit are often specified as a mass loading. To determine a mass loading and thereby evaluate compliance with permit limits, it is necessary for the sampler to obtain accurate flow data. Flow measurement is the commonly used term for this process. In addition to verifying compliance with permit limits, flow measurement serves to:

a. Provide operating and performance data on the wastewater treatment plant

- b. Compute treatment costs, based on wastewater volume
- c. Obtain data for long-term planning of plant capacity.

Specific operating instructions for automatic samplers, capabilities, capacities, and other pertinent information are included in the respective operating manuals and are not presented here.]]

4.2.4.3 Sample Types

There are two primary types of samples: 1) grab samples; and 2) composite samples. Each type has distinct advantages and disadvantages. In order to obtain a more complete characterization of a specific facility's effluent, the two sample types can be used independently or in combination.

a. Grab Samples

1. This is an individual sample collected over a period of time, usually all in one motion, generally not exceeding 15 minutes. The 15 minute time limit applies to aqueous samples only. No particular time limit applies to the collection of solid samples (e.g. residuals). Grab samples may be used to determine consistency between an industry's self-monitoring data and to corroborate the results of composite samples.

2. Grab samples represent the conditions that exist at the moment the sample is collected and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

- a. A snapshot of the wastewater quality at a particular instant in time is desired; and
- b. The water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow);
- c. The characteristics of the water or waste stream are known to be constant or nearly so;
- d. When the waste conditions are relatively constant over the period of discharge. In lieu of complex sampling activities, a grab sample provides a simple and accurate method of establishing waste characteristics;
- e. The sample is to be analyzed for parameters whose characteristics are likely to change significantly with time (i.e., dissolved gases, bacteria, pH, etc.);
- f. The sample is to be collected for analysis of a parameter such as oil and grease or bacteriologicals

where the compositing process could significantly affect the actual concentration;

g. Data on maximum/minimum concentrations are desired for a continuous water or wastewater stream; and

h. Identifying and tracking slug loads and spills.

3. If required to be measured, the following parameters shall be measured on grab samples or in-situ. NOTE: If the permit specifies a composite sample for any of the above-mentioned parameters, THE PERMIT CONDITIONS SHALL BE FOLLOWED.

pH	phenol
temperature	oil and grease
dissolved oxygen	bacterial
sulfide	volatile organic compounds
chlorine residual	specific conductance
other dissolved gases	cyanide
un-ionized ammonia	dissolved constituents in field filtered samples (ortho-P, metals, etc.)

4. Sampling protocols shall follow those outlined under Surface Water (4.2.3.3.a).

b. Composite Samples

1. A composite sample is a sample collected over time, formed either by continuous sampling or by mixing discrete samples. Composite samples reflect the average characteristics during the compositing period.

2. Composite samples are used when stipulated in a permit and when:

- a. The water or wastewater stream is continuous;
- b. Analytical capabilities are limited;
- c. Determining average pollutant concentration during the compositing period;
- d. Calculating mass/unit time loadings; and
- e. Associating average flow data to parameter concentrations.

3. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus ten percent of the average flow rate, or they may be collected proportional to the flow rate. The permit may specify which composite sample to use, either time composites or flow proportional composites. The compositing methods, all of which depend on either continuous or periodic sampling, are described in the following discussions.

4. Time Composite Sample

- a. Time composite samples are based on a constant time interval between samples.

b. A time composite sample can be collected manually or with an automatic sampler.

c. This type of composite is composed of discrete sample aliquots collected in one container at constant time intervals.

d. This method provides representative samples when the flow of the sampled wastewater stream is constant. This type of sample is similar to a sequential composite sample (described below).

5. Flow Proportional Composite Sample

a. Flow proportional samples can be collected automatically with an automatic sampler and a compatible pacing flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually.

b. There are two methods used to collect this type of sample:

1. One method collects a constant sample volume per stream flow (e.g., 200 milliliters (ml) sample collected for every 5,000 gallons of stream flow) at time intervals proportional to stream flow. This method provides representative samples of all waste streams when the flow is measured accurately. For this reason, it is used frequently.

2. In the other method, the sample is collected by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots (e.g., hourly samples are taken with the sample volume being proportional to the flow at the time the sample is taken).

6. Sequential Composite Sample - Composed of discrete samples taken into individual containers at constant time intervals or constant discharge increments.

a. For example, samples collected every 15 minutes are composited for each hour.

b. The 24-hour composite is made up from the individual one-hour composites.

1. Each of the 24 individual samples is manually flow proportioned according to the flow recorded for the hour that the sample represents.

2. Each flow proportioned sample is then added to the composite samples.

3. The actual compositing of the samples is done by hand and may be done in the field or the laboratory.

4. In most cases, compositing in the field is preferable since only one sample container must be cooled, and then transported to and then handled in the laboratory.

5. A 24-hour composite is frequently used since an automatic sampler can easily collect the individual samples.

c. A variation of the 24-hour composite is to collect a constant volume of sample taken at constant discharge increments, which are measured with a totalizer. For example, one aliquot is collected for every 10,000 gallons of flow.

d. Sequential sampling is useful to characterize the waste stream because you can determine the variability of the wastewater constituents over a daily period.

For example, for pretreatment studies you can visually determine when high strength wastes are being discharged to a facility or when heavy solid loads are being discharged during a 24-hour cycle. You can measure different pHs throughout the day. The value of this type of sampling must be weighed against the manpower constraints and sampling goals.

7. Continuous Composite Sample - Collected continuously from the waste stream. The sample may be a constant volume which is similar to the time composite, or the volume may vary in proportion to the flow rate of the waste stream, in which case the sample is similar to the flow proportional composite.

8. Areal Composite - A sample composited from individual grab samples collected on an areal or cross-sectional basis. Areal composites shall be made up of equal volumes of grab samples; each grab sample shall be collected in an identical manner. Examples include soil or residual samples from grid system points on a land application site, water samples collected at various depths at the same point or from quarter points in a stream, etc.

4.2.4.4 General Concerns

a. The sampler must weigh advantages and disadvantages when choosing between the use of grab or composite sampling methods.

1. While grab sampling allows observation of unusual conditions that may exist during discharge, such as sudden bursts of color or turbidity, this method is labor intensive and impractical when sampling is performed at many locations over extended periods of time.

2. When sampling a large number of locations, the use of automatic samplers is more practical.

a. Automatic samplers also help reduce human error, specifically in complex sampling activities, such as flow proportional sampling, and reduce exposure to potentially hazardous environments.

b. The primary disadvantage to automatic sampling is the cost of the equipment and maintenance requirements. Many automatic samplers in use today are electronically controlled and must be sent back to the manufacturer when a malfunction occurs.

b. In order to obtain a representative sample, sampling must be conducted where wastewater flow is adequately mixed. In general these criteria shall be used to evaluate the location:

1. A sample should be taken in the center of the flow where velocity is highest and there is little possibility of solids settling.

a. The sample should be collected at a depth between 40% - 60% of the total depth where the turbulence is maximized. This means that sample collection should be avoided at the water surface or the channel bottom.

b. Flow mixing is particularly important for ensuring uniformity.

2. Sampling personnel should be cautious when collecting samples near a weir because solids tend to collect upstream and floating oil and grease accumulate downstream.

3. If the sample is not to be tested for volatile organics or will not be affected by stripping of dissolved gases, mechanical stirring may be used or a stream of air may be introduced into the waste stream.

4. In sampling from wide conduits, cross-sectional sampling should be considered. Dye may be used as an aid in determining the most representative sampling point(s). Note: appropriate Department personnel should be consulted for the type of dye and acceptable protocols.

5. If manual compositing is employed, the individual sample bottles must be thoroughly mixed before pouring the individual aliquots into the final composite container.

6. If the sample is taken from an effluent tap, allow the tap to run for one - two minutes to allow the settled solids to flush from the line. Reduce the flow to 500 ml/min before collecting the samples.

c. Sampling and flow measuring are integrally related. The sampler must know the wastewater flow variability before a sampling program can be initiated. Whether to use a flow proportional or time composite sampling scheme depends on the variability of the wastewater flow. If a sampler knows or suspects significant variability in the wastewater flow or knows nothing about the facility, a flow proportional sample should be collected; otherwise a time composite sample would be acceptable.

d. Prior to sampling, the flow measuring system (primary flow device, totalizer, recorder) should be examined (see sections on Stage and Flow Measurement in the EPA SOP, Sections 7.6 and 7.8). If the flow measuring system is unacceptable, the sampler may have to install a flow measuring device. If the flow measuring system is acceptable, samples can be collected by the appropriate method.

e. Fill out the information on the sample container tags and on the field sheets completely and carefully. Improper sample identification results in invalid or unacceptable samples and lost sampling efforts.

f. Take inordinate care to prevent cross-contaminating samples. Use properly cleaned sampling equipment.

4.2.4.5 Sample Equipment Requirements

a. Manual Sampling

1. The types of sampling devices that may be used to collect samples are specified in the next section. Additional discussions are found in the protocols for collection grab samples in surface water (section 4.2.3.3.a).

2. IN ALL CASES, the selected sampling equipment shall be compatible with the components to be collected and shall comply with the use and construction restrictions specified in Table 4.1.

3. All equipment shall be cleaned using the appropriate protocols specified in Section 4.1. Sample containers shall be cleaned according to Section 4.4.1 or obtained precleaned from commercial sources.

b. Automatic Samplers

1. A wide variety of automatic samplers are commercially available (e.g., Sigma, ISCO). Most have the following five interrelated subsystem components:

a. Sample Intake Subsystem - The sample intake gathers representative samples from the sampling stream.

1. The intake is usually the end of a plastic suction tube which should also be resistant to physical damage from large objects in the flow stream. Nonleaching tygon tubing is most often used.

2. Teflon tubing shall be used under the conditions specified in 4.2.4.5.b.2.b. The end of this tubing should be fixed to a piece of conduit or a pole bent to hold the sample port in the waste stream at the correct location to get a

representative sample. The tubing shall be supported in such a way that the incoming sample is not contaminated by either the supporting pole or the method of attachment.

b. Sample Gathering Subsystem - Automatic samplers provide one of three basic gathering methods:

1. Mechanical - Mechanical gathering subsystems are usually built into place and include devices such as wide/deep channel flow. Because of the mechanical aspects on cables, calibrated scoops, and paddle wheels with cups. Although these systems obstruct the stream flow, they take into account site specific considerations, such as high sampling lift and system employed, these units require periodic maintenance.

2. Forced Flow - Forced flow gathering subsystems are often built into place as permanent sampling facilities; thus, like the mechanical gathering subsystems, they may obstruct the stream flow. They also require periodic inspection and maintenance. However, forced flow subsystems have the advantage of being able to sample at great depths. In addition, because this gathering system uses air pressure to transport the sample, it may be ideal for sample collection in potentially explosive environments.

3. Suction Lift - The suction lift is the most widely used type of sample gathering subsystem due to its versatility and minimal affect on flow patterns. Suction lifts are limited to 25 vertical feet or less because of internal friction losses and atmospheric pressure. At least 100 ml should be collected each time the pump is actuated.

c. Sample Transport Subsystem - The sample is usually transported from the sample intake to the collection bottle by a plastic tube referred to as the sample transport subsystem. Care should be exercised to avoid sharp bends and twists in the transport line.

d. Sample Storage Subsystem - The sample storage subsystem can accommodate either a single large collection bottle or a number of smaller collection bottles.

1. The total sample volume storage capability should be at least 2 gallons (7.6 liters): some samplers have a capacity as great as 5 gallons.

2. To thermally preserve the samples, storage subsystems must be large enough to provide space for ice to chill the sample during collection (see b.3 below).

3. Samples with individual bottles for discrete collection are usually equipped with a cassette which

rotates to fill the bottle at the time of sampling.

e. Controls and Power Subsystem

1. The control units allow selection of time or flow compositing method, or continuous sampling method. The automatic samplers most widely used have encapsulated solid state controls. This minimizes the effects of unfavorable environments that may be encountered in the field, such as high humidity and corrosiveness. These units are also sealed so that they may be used with minimum risks in potentially explosive environments. In addition, sealed units protect the controls if the sampler is accidentally submerged.

2. Samplers operating from a power supply are more reliable than battery operated models; however, field conditions often prohibit the use of a power supply.

2. Automatic sampling equipment must meet the following requirements:

a. Sampling equipment must be properly cleaned to avoid cross contamination which could result from prior use (see section 4.1.5 for specific cleaning procedures).

b. If samples for organics (includes all extractable organics, pesticides, and herbicides and TOC) are to be collected, no plastic or non stainless steel parts of the sampler shall come in contact with the water or wastewater stream:

1. Teflon tubing shall be used in the transport subsystem.

2. A special sampler base and glass containers may also be necessary for sampling organics. Consult your owner's manual.

c. If the preservation requirements for a particular component specify that a sample be thermally preserved, the sampler must be able to keep the samples cool to 4 C during the sampling period. This is accomplished in the field by using ice or refrigeration units in the sampler.

d. The sampler must be able to collect a large enough sample for all parameter analyses. Additionally, split samples may also be necessary.

e. A minimum of 100 ml should be collected each time the sampler is activated, if a peristaltic pump is used.

f. The sampler should provide a lift up to at least 20 feet and the sampler should be adjustable so that volume is not a function of the pumping head.

g. The pumping velocity must be adequate to transport solids and not allow solids to settle.

h. Pump intake line

1. The automatic sampler must provide for line purging after each sample is drawn to prevent contamination of subsequent samples.

2. The minimum intake line inside diameter shall be at least 1/4 inch, which is large enough to lessen chances of clogging but small enough to maintain velocity and to avoid solids settling.

i. Sample transport system

1. The tubing should be at least 1/4 inch inside diameter to maintain adequate flow and to prevent plugging.

2. Tubing should be sized so that a velocity of at least two feet per second can be maintained.

3. Line must be automatically purged after each sample is collected.

10. An adequate power source should be available to operate the sampler for 48 hours at a 30-minute sampling interval.

j. Sample collection vessels large composite or discrete sample containers, shall be constructed of materials appropriate for the tests to be performed. As a general rule, the vessels shall be made of the same material as those specified for sample containers in section 4.4.2.3.

3. In addition to the requirements listed in 4.2.4.5.b.2 above, several factors should be considered in selecting automatic sampling equipment. Among these are:

a. Convenience of installation and maintenance - Sampling equipment should always be handled carefully and maintained in accordance with the manufacturer's instructions. Most equipment failures are caused by careless handling and poor maintenance.

b. Equipment security

1. Security is important when sampling is done as part of an enforcement proceeding.

2. Manhole locations where battery operated equipment may be installed and the cover replaced will aid in maintaining security.

3. If sampling equipment must be left unattended, the sampler should be provided with a lock or seal which, if broken or disturbed, would indicate that tampering had occurred.

c. Operation in cold or hot weather

1. Cold weather - In Florida, use of automatic samplers is seldom a problem during cold weather. If

a sample must be taken during extremely cold days freezing of intake lines may happen. These problems may be handled by using heat tape or placing the sampler inside a thermostatically controlled, electrically heated enclosure. In the absence of special equipment, freezing may be prevented by installing the sampler in a manhole or wet well or by wrapping the sampler with eight or nine inches of insulation and wind protection. Also, the sampler should be positioned well above the effluent stream so that the tubing runs in a taut, straight line to prevent pooling of liquid.

2. Hot weather - The summer heat in Florida does pose a significant problem with keeping the sample cool. If possible, choose a shaded or even cooled place for the sampler. If not, insulation wrapped around the sampler may help. Painting the sampler white will reflect some heat. Before leaving the site refill the automatic sampler with ice or check to see that the refrigeration unit is operating. An attempt should be made to pick up the samples near the time the last sample is taken. Samples which are allowed to warm up are questionable, if not useless.

4.2.4.6 Sample Acquisition

a. Manual Sampling

1. Manual sampling is used for collecting grab samples for immediate in-situ field analyses. However, it can also be used in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to observe and/or note unusual waste stream conditions.

2. Collection using the Sample Container

- a. The actual sample container must always be used for collecting samples for oil and grease, volatile organic compound (VOC), and bacteriological samples.
- b. If possible, manually collected samples should be collected in the actual sample container that will be submitted to the laboratory. This eliminates the possibility of contaminating the sample with an intermediate collection container.
- c. Sample containers containing premeasured amount of preservatives SHALL NOT be used to collect surface water grab samples. Alternative collection procedures listed below must be followed.
- d. Sample collection shall follow the protocols for collecting simple grab samples in surface water (Section 4.2.3.3.a).

3. Sampling with an intermediate vessel or container
 - a. If the sample cannot be collected as described above, an intermediate vessel can be used.
 - b. The sample shall be collected following the protocols described for collecting surface water grab samples with an intermediate container or pond sampler (Section 4.2.3.3.a) and redistributed into appropriate sample container(s).
 4. Samples collected in bailers
 - a. Bailers may be used if the data requirements do not necessitate a sample from a strictly discrete interval of the water column.
 - b. Bailers with an upper and lower check-valve can be lowered through the water column and water will be continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. This technique may not be successful in strong currents.
 - c. Specific sampling protocols outlined under mid depth surface water sampling (4.2.3.3.b.4) shall be followed.
 5. Samples collected with pumps
 - a. In some cases it may be best to use a pump, either power or hand operated, to withdraw a sample from the water or wastewater stream.
 - b. Protocols for the use of pumps are specified in the surface water sampling Section (4.2.3.3.b.5) and shall be followed when collecting with pumps.
 6. Dedicated equipment may also be used at each sampling station. This will avoid cross contamination between sampling stations. For most parameters (other than trace metals and organics) rinsing the sampling device three times in the effluent stream is sufficient. More stringent requirements must be used for trace pollutants (see Section 4.1).
- b. Automatic Samplers
1. Automatic samplers may be used when several sites are to be sampled at frequent intervals or when a continuous sample is required.
 2. Conventional Sampling: All composite samplers can be used to collect time composite or flow proportional samples.
 - a. In the flow proportional mode, some samplers are activated by a compatible flow meter.

- b. Flow meter operation will not be discussed here. Refer to the operating manuals if you have them.
- c. For older models, flow proportional samples can be collected using a discrete sampler and a flow recorder and manually compositing the individual aliquots in flow proportional amounts.

3. Installing the Composite Sampler

a. All new or precleaned tubing (Dow Corning Medical Grade Silastic, or equal, in the pump and either Teflon or Tygon, depending on the parameters of interest, in the sample train) shall be used each time the sampler is installed.

1. Cut the proper length of precleaned Teflon or Tygon tubing;

2. Rinse deionized water through the sampler and collect an equipment blank;

3. Put the collection sieve and tubing in the appropriate sample location in the wastewater stream, using conduit if necessary to hold it in place. Assure that the incoming sample water is not contaminated by the supporting conduit; and

4. Program the sampler as per manufacturer's directions and as required in the permit conditions.

b. For a time composite sample, the sampler should be programmed to collect 200 ml at 30-minute intervals or 100 ml at 15-minute intervals into a refrigerated 3-gallon jug. For a 5-gallon compositing jug, the volume should be increased accordingly.

c. For a flow proportional sample, the sampler should be programmed to collect a minimum of 100 ml for each sample interval, with the interval predetermined based on the flow of the waste stream.

d. At the end of each 24-hour sampling period, the contents of the compositing jug (sample) should be stirred and siphoned (poured if no visible solids) into the respective containers, followed by immediate preservation, if required.

4. Automatic Sampler Security - A lock or seal may be placed on the sampler to prevent or detect tampering. However, this does not prevent tampering with the sampler tubing (see additional discussions on sample security under equipment requirements 4.2.4.5.b.3 above)

5. Sample Preservation - Samples shall be preserved for all samples according to 40 CFR Part 136 Table II.

a. Table II includes allowances for automatic samplers. In addition to the capability of keeping

samples cooled with ice or refrigeration, there are 2 considerations to be presented for chemical preservation:

1. If separate bottles are used, they may be prepreserved with the appropriate chemical preservative or preserved after sampling has been completed (WITHIN 24 HOURS);
2. If the large compositing jug is used, preservation should be completed after sampling has been completed (WITHIN 24 HOURS);
3. NOTE: If the only parameter of interest is Total Phosphorus, and the project is unrelated to a NPDES permit, then the sample must be chemically preserved (H_2SO_4) but it need not be cooled to 4 C with wet ice. The acid must be in the container prior to sample collection.

4.2.5 Groundwater Sampling

4.2.5.1 Introduction and Scope

This section presents the standard operating procedures that should be employed during field investigations to ensure that representative groundwater samples are collected. The importance of proper sampling of monitor wells cannot be overemphasized. Care should be taken so that the sample collected is neither altered nor contaminated by sampling and handling procedures.

The following discussions cover acceptable: equipment choice, equipment construction materials, pre-sampling and in-field decontamination, purging and sampling technique, and proper field Quality Control procedures. Although not a complete discussion of all groundwater sampling procedures, this information has been compiled with the intent of providing the equipment and techniques for situations that are most likely to be encountered.

[[4.2.5.2 Selection of Sampling Materials

This section is intended as a GUIDE to be used when selecting sample collection and/or well purging equipment. The discussions are informational and should be considered as criteria when selecting equipment.

The selection of inert materials used for well purging, sample collection, handling and storage is a critical consideration in planning the well-conceived cost-effective monitoring program. These equipment descriptions may also be used for other sampling matrices. The materials of choice should retain their structural integrity for the duration of the monitoring program. They must

not react with the sample (i.e., should not absorb, adsorb or leach sample constituents) which would bias representative sample collection. Additionally, the sampling equipment should be chosen so that they can be easily decontaminated and transported. The following subsections, adapted from Barcelona, et al. (1983) and Watts (1988), discuss sampling material construction.

a. Stainless Steel - The most common types of stainless steel that are used in sample collection equipment are 316 and 304 although other types are available and may be used.

1. Stainless Steel-316

a. Recommended for use in most groundwater and soil monitoring situations for all parameter groups.

b. Stainless Steel-316 should be utilized especially for detailed organic and trace metals analytical work.

c. Stainless Steel-316 is least likely to introduce sampling bias or imprecision.

d. Stainless Steel-316 is also relatively easy to decontaminate without requiring a HNO₃ rinse.

e. The cost per foot of Stainless Steel-316 is approximately the same as Teflon.

f. Note that dedicated stainless steel sampling equipment (tubing, submersible pump housing, etc.) may not be suitable to collect samples for metals analyses since its performance may be sensitive to the chloride ions, which can cause pitting and corrosion over long term exposure under acidic conditions.

2. Stainless Steel-304

a. Recommended for use in most groundwater and soil monitoring situations for all parameter groups (including trace organics and metals).

b. Stainless Steel-304 is less corrosion-resistant than 316 and may be prone to show pitting and corrosion if left in contact with acidic high total dissolved solids (TDS) groundwaters for extended periods (dedicated downhole equipment).

c. Corrosion products are mainly limited to iron and potentially Cr, Cu and Ni. Pitted surfaces may present active adsorption sites and render difficulty in future decontamination.

c. Teflon

1. Teflon is the trademark of Dupont, Inc. Teflon is recommended for use in most groundwater and soil monitoring situations for all parameter groups, especially for detailed, trace organic analytical work.

2. Teflon material is least likely to introduce sampling bias or imprecision. Teflon is relatively easy to

decontaminate. Teflon is a relatively soft material and is easily scratched.

3. Bailers and tubing that are old and excessively scratched will be difficult to clean and must be discarded.

4. Some Teflon bailers have replaceable "donuts" that encircle the bailer and protect the body from scratches.

d. Low-Carbon Steel, Galvanized Steel and Carbon Steel

1. These materials are generally not recommended for collecting samples, especially galvanized steel.

2. In cases of split spoon and core barrel soil sampling these might be the only material available.

a. If trace metals are of interest, plastic or teflon liners shall be used to prevent the sample from contacting the equipment surface.

b. Teflon liners are recommended if organics are of interest.

3. New equipment made from these materials must be very carefully cleaned to remove oily manufacturing residues.

4. Corrosion is likely in high dissolved solids and acidic environments, particularly when sulfides are present.

5. Products of corrosion for carbon steel are mainly Fe and Mn, except for galvanized steel which may release Zn and Cd.

6. Weathered steel surfaces present very active adsorption sites for trace organic and inorganic chemical species and pitted surfaces will increase the difficulty of decontamination.

e. Polypropylene/Polyethylene

1. Polypropylene and polyethylene are polyolefin materials that are more resistant to organic solvent attack than formulated plastics such as Viton, silicone or neoprene.

2. Polypropylene and polyethylene are comparable in performance and resistance to Teflon in corrosive high dissolved solids (Pettyjohn et al., 1981 and Barcelona et al., 1983).

3. Polypropylene and high density polyethylene (HDPE) tubing material may be used for purging wells (see Table 4.1 for details).

4. HDPE disposable bailers may be used to sample all parameters except organic compounds.

f. Polyvinyl chloride (PVC)

PVC (flexible) is not recommended for detailed organic analytical work. The polymer formulated PVC contains plasticizers, stabilizers and antioxidants which may cause

interferences with analytical determinations, especially when industrial solvents are encountered in groundwater. Documented interferences are likely with several priority pollutant classes.

g. Viton, Tygon, Silicone and Neoprene

Viton, Tygon, silicone and neoprene are not recommended for organic analytical work since the inherent plasticizers, stabilizers and antioxidants may introduce interferences.]]

4.2.5.3 Purging and Sampling Equipment

a. General Considerations

1. Purging the monitor well of stagnant water can be performed with various equipment. The choice of equipment will depend on the parameters of interest, the well diameter, the well specific capacity, transmissivity, the water level elevation and other site conditions. As stated earlier, the choice of equipment used for purging must not bias the "representativeness" of the sample collected.

2. It is recommended that field personnel use pumps to purge monitor wells if at all possible.

3. Bailers are not recommended for purging monitor wells because frequent lowering and retrieving of the bailer:

- a. will introduce atmospheric oxygen which may precipitate metals (e.g. iron) or cause other changes in the chemistry of the formation water (i.e. pH),
- b. will result in agitation or volatilization of groundwater which may bias volatile and semi-volatile analyses, and
- c. may introduce dirt through scraping the sides of the casing wall.

4. Though bailers are not recommended for purging, they are acceptable if constructed of the appropriate material and if extreme care is used. The use of bailers is described in 4.2.5.3.c below.

5. All standing water around the top of the well casing (manhole) shall be removed before opening the well.

b. Pumps - A summary of the principles of operation and the advantages and disadvantages of the various commercial pumps is given in "Monitor Well Construction and Groundwater Sampling" (Watts, 1988).

1. Above-ground Pumps

- a. Peristaltic Pump - Peristaltic pumps may be used to purge low volume, low specific capacity wells in

which the static water level in the well is no greater than 20-25 feet BLS (Below Land Surface).

1. Decreased pumping velocity will be experienced when water levels are deeper than 18'-20'.

2. It also may be used to sample wells for limited parameter groups. These parameter groups will be dependent upon tubing materials and arrangements. It is the preferred method of collecting filtered groundwater samples for metals. See Table 4.1 for details on the restrictions for this pump, including choice of tubing (i.e. Teflon, HDPE, Tygon). See EPA Region IV SOP & QAP, Appendix F.1 for additional guidance.

b. Centrifugal Pump - Centrifugal pumps can be utilized to purge 2 inch and larger internal diameter wells that have moderate specific capacities from 2 - 10 gpm (gallons per minute) and have a static water level greater than 20 feet BLS.

1. The pump may also be attached directly to 3/4" well point casing and used to purge (see 4.2.8, care must be taken so that purged water does not fall back into the well casing).

2. Sampling gloves shall be worn and discarded after positioning the pump. Hands should be washed and new gloves shall be put on prior to sampling.

3. See Table 4.1 for compatibility restrictions related to choice of tubing and allowable parameter groups.

2. Submersible Pumps

a. Electric Submersible Pumps - Submersible pumps (e.g. Grundfos, Goulds, Jacuzzi) can be utilized for purging 4 inch or greater diameter monitor wells. Some submersible pumps can be utilized in 2 inch wells (e.g. Fultz and Grundfos). These pumps can be used in wells that have moderate to high specific capacity and cannot be purged using an above-ground pump because of the lower static water level elevation (>20'-25' BLS).

1. The pump must be constructed of stainless steel (and/or Teflon) material and the delivery hose shall be constructed of appropriate material depending upon the analytes of interest.

2. It may be fitted with inert stainless steel or Teflon tubing between the pump and "other non-inert tubing" to be able to purge wells that will be sampled for trace organics.

3. See EPA Region IV SOP & QAM, Appendices F.2-F.3 for further information regarding 2 and 4 inch electric, submersible pumps.

b. Bladder Pumps - Positive-displacement bladder pumps (no-gas contact) can be utilized for purging wells where the water table is greater than 25 feet and an above-ground pump cannot be used. These pumps are used in wells with low to moderate capacity since pumping rates are not as high as the electric submersibles or the gas-contact "purge pump" described below. Maximum pumping rates are approximately 0.5 - 1.5 gallons per minute depending upon the location of the pump (BLS).

1. The bladder pump system is composed of three major components: the pump, the compressed air and water discharge tubing, and the controller/compressor.

2. The pump consists of a bladder and an exterior casing or pump body that surrounds the bladder. These two parts can be composed of various materials, usually combinations of PVC, Teflon, and stainless steel.

3. The construction material of the pump body, pump bladder, and the discharge tubing will define the parameters that can be purged and sampled with this system.

4. If the pump is not permanently installed in the monitor well and if it is to be used to purge and/or sample for all parameters (including VOCs), the pump, bladder and tubing must be constructed of stainless steel and Teflon.

5. Permanently installed pumps have a PVC pump body as long as the pump remains in contact with the formation water. If VOCs and/or extractable organics are of interest, the bladder and the delivery tubing shall be constructed of teflon.

c. Bladderless Purge Pumps - These pumps are identical to the bladder pumps described above except they do not have an internal bladder. The air controller/compressor is used to force water from within the pump body up the discharge tubing. By not having the (Teflon) bladder fill by head pressure, pumping rates are much higher (>4 gpm).

1. This pump can only be used for purging.

2. Additionally, operation of this pump cannot result in purge water escaping back into the well. Proper operation and maintenance of the check valve

must be ensured. Release of aerated purge water into the water column is not acceptable.

3. Hand Pumps

a. Hand pumps (e.g. Brainard-Kilman 'B-K Pump') are manual pumps that should be utilized for purging 2- or 4-inch diameter monitor wells in which the static water level is too deep for use of a centrifugal or peristaltic pump.

1. The B-K hand pump and the associated riser pipes are constructed of PVC and shall be used to purge when only inorganic constituents are of interest unless the restrictions specified in Table 4.1 are followed.

4. The lower most section of the B-K pump is equipped with a foot valve to prevent back flow of purge water.

5. After purging has been completed, the B-K pump should be completely disassembled and decontaminated.

6. Please see Table 4.1 for details on the use of this pump.

c. Bailers

1. As stated above, the use of bailers is not recommended for purging.

2. Bailers shall be composed of material compatible with the analytes of interest. See Table 4-1 for restrictions

a. Bailers constructed of stainless steel and Teflon may be used to sample any and all parameters.

b. Bailers constructed of high density (rigid) polyethylene (HDPE) materials may be used to sample monitor wells for inorganics and free-product only.

c. When sampling grossly contaminated tanks or other facilities, disposable polyethylene (or other material) bailers should be utilized (it may be difficult to decontaminate such grossly contaminated bailers and as such they may have to be discarded).

3. The bailer must be handled carefully so as not to contaminate it prior to use.

4. They shall be scrupulously cleaned, including all check valves (see Section 4.1.4.1).

d. Lanyards

1. Lanyard may be disposable (braided or monofilament nylon or reusable (stainless steel or teflon-coated)).

2. A disposable lanyard must be changed for each monitor well, but the same lanyard may be used for purging

(if performed) and sampling operations without decontamination between purging and sampling operations.

3. Reusable lanyards shall be decontaminated between monitor wells (see Section 4.1.9.1) but do not require cleaning between purging and sampling operations.

4.2.5.4 Water Level and Purge Volume Determination

Prior to sampling, an adequate amount of stagnant well water in the well must be removed in order to sample representative formation water.

a. Inspect the exterior protective casing monitor well for damage and document accordingly.

b. Water Level Measurements

1. In order to calculate the purge volume, the water level is determined by using an electronic probe, chalked tape, etc.

2. The depth below land surface shall always be recorded to the nearest 0.1 foot from the same reference or survey mark on the well casing.

3. Measurements using an electronic probe shall follow the manufacturer's instructions. Since false reading may be obtained if the sensor contacts the well casing, multiple readings shall be taken to ensure accuracy.

4. Chalked Tape Method

a. Lower chalked tape into the well until the lower end is in the water (usually determined by the sound of the weight hitting the water).

b. Record the length of the tape relative to the reference point (see 2 above).

c. Remove tape and note the length of the wetted portion.

d. The depth to water is determined by subtracting the length of the wetted portion (c above) from the total length (b. above).

5. Decontaminate all measuring devices immediately after use (see 4.1.9.1) and prior to next measurement.

c. Water Column Determination

1. The total water column is obtained by subtracting the depth to the top of the water column from the total depth of the well.

2. Total depth of well is dependent upon the well construction. Some wells may be drilled in areas of sinkhole or karst formations. In cases where there may be an open borehole below the cased portion, an attempt should be made to find the total borehole depth.

d. Well Water Volume

The length of the water column is then converted to volume of water that is present in the well:

1. 2 inch casing:

$$V = 0.17 \times h$$

Where: V = volume in gallons

h = height of the water column in feet

2. 4 inch casing:

$$V = 0.66 \times h$$

Where: V = volume in gallons

h = height of the water column in feet

3. For other casing sizes, calculate using the following:

$$V = (0.041)d \times d \times h$$

Where: V = volume in gallons

d = well diameter in inches

h = the height of the water column in feet

or:

$$V = \pi \times r \times h(0.001)$$

Where: V = volume in liters

P = 3.14159 (pi)

r = radius in centimeters

h = height of water column in centimeters

e. Record all measurements in the field records.

4.2.5.5 Well Purging Techniques

To ensure a representative groundwater sample from a monitor well it is essential that the well be purged prior to sampling. Stagnant water in a well casing may undergo a variety chemical changes due to alterations in the redox potential, pH and leaching of organic compounds from the casing.

Several methods of purging wells have been cited in the literature (Gibb et al., 1981, and Barcelona et al., 1983). The choice of purging technique may be dictated by hydrogeologic properties (particularly depth to water table and hydraulic conductivity).

a. Equipment selection shall comply with construction and configuration requirements specified in Table 4.1.

b. A clean protective covering may be placed around the wellhead during purging activities. If this protective covering becomes soiled, ripped, etc. it must be replaced prior to sampling.

c. The total amount of water must be recorded. Therefore, the volume must be measured during the purged operation. The amount may be determined by:

1. Collecting the water in a graduated container (i.e. bucket); or
 2. Calculating volume based on pumping rate. Note: the pumping rate may not be constant; take this into account.
- d. Record the time that actual purging begins in the field records.
- e. Purging is considered complete if any one of these criteria are satisfied:
1. three well volumes and subsequent stabilization of field parameters
 - a. Stabilization of field parameters is defined as "consecutive readings within 5% taken at least five minutes apart".
 - b. Even if field parameters have not stabilized after 5 well volumes, purging is considered complete and sampling can begin.
 2. five well volumes (field parameters not monitored);
 3. at least one fully dry purge.
 - a. It has been suggested that one dry purge may not be adequate and a second dry purge may be necessary. Theories concerning purging the sandpack or aeration of sandpack will not be discussed here.
- f. Except for "low recovery" wells, all wells shall be sampled within 6 hours of purging.
1. "Low recovery" wells or wells that have been purged complete dry may be sampled as soon as sufficient sample matrix is available or up to 10 hours after purging.
 2. Wells that have not recovered sufficiently within 10 hours of purging should not be sampled.
- g. Lanyards
1. All lanyards must be securely fastened to downhole equipment (bailers, pumps, etc.).
 2. Equipment construction and decontamination shall follow guidelines discussed in Purging and Sampling Equipment above (4.2.5.3.d).
 3. Bailer lanyards must be handled such that they do not touch the ground surface.
- h. Low Hydraulic Conductivity Monitor Wells (i.e. wells that can be purged dry)
1. The most straightforward method for removing all of the stagnant water in wells screened in low hydraulic conductivity formations is to install the pump in the screen area and pump the well dry.

2. Although this procedure may allow the atmosphere to contact the area of the aquifer immediately surrounding the well screen, it is the best way to ensure that all the stagnant water has been removed.

3. If required, allow the well to recover and purge the well a second time.

i. High Hydraulic Conductivity Wells (i.e. wells that cannot be purged dry)

1. For those wells with dedicated purging/sampling systems where the pump is set in the screened area of the well, complete evacuation of the stagnant water column may not be possible.

2. The degree to which the stagnant water column can be replaced by fresh aquifer water will be a function of the aquifer transmissivity and the number of well volumes pumped (Barcelona et al., 1983).

3. If in doubt, a short pump test or slug test may be performed on each monitor well and the number of well volumes calculated to assure replacement of the stagnant water.

j. In general, when nondedicated pumps that are used for purging, the purging process should be done with the pump as near to the top of the water column as possible to ensure that no stagnant water remains in the well above the screen after purging.

k. Peristaltic Pump - One end of a length of new or pre-cleaned tubing shall be attached to the pumphead flexible hose and the other end immersed no deeper than one foot into the water column.

l. Centrifugal Pumps

1. To minimize cross contamination while purging, fuel driven centrifugal pumps must be placed at least 10 feet from the well head and downwind of the well.

2. Sampling gloves shall be worn and discarded after positioning the pump. Hands should be washed and new gloves shall be put on prior to sampling.

3. The length of suction hose should be situated such that the pump will be withdrawing water from the top of the column.

4. If the pump rate exceeds the recovery rate of the well then the hose should be lowered into the well as needed to accommodate the drawdown.

5. The suction hose must have a footvalve installed to prevent purge water from re-entering the well.

m. Electric Submersible Pumps

1. The pump should be set as near the top of the water column as possible to ensure that all stagnant water in the casing is removed and to minimize the contact area of the delivery hose with water column.

2. If the pump rate exceeds the specific capacity of the well then the pump must be lowered to accommodate the drawdown.

3. If the pump has a controller, the flow rate may be adjusted to be equal (or nearly) to the well's pumping capacity.

n. Bladder Pumps

1. This equipment shall be operated strictly according to the owners/operators manual or sample integrity and representativeness may be suspect.

2. After determining water level, position the controller/compressor away from the well and downwind (if fuel powered compressor or generator).

3. Attach tubing and lower the pump to a depth of 3 - 5 feet below the surface of the water.

4. If the pump is positioned too deep all of the stagnant water may not be purged. If positioned too shallow purging time will be slower as the bladder fills under standing head pressure.

5. Adjust the pump position to follow the water level drawdown, if necessary.

6. It may be necessary to adjust the purging rate so that it is equivalent to the drawdown rate.

7. Discharge must be directed into graduated bucket or equivalent to determine the number of well volumes.

o. BK Hand Pump

1. For the B-K Pump, the intake is lowered to the top of the water column by attaching additional 5-foot sections onto the pump.

2. By changing the stroke of the actuating rod the pumping rate can be made compatible with the well-specific yield.

p. Bailers

1. The bailer must be handled carefully so as not to contaminate it prior to use.

2. The bailer must be lowered through the well and into the formation water slowly. Allowing the bailer to drop into the formation water with a splash is not acceptable.

3. The bailer should be used to pull purge water from the top of the water column so that fresh aquifer water can

be pulled in through the screen. This technique shall be performed until the requisite number of well volumes have been evacuated.

q. All purging activities shall be documented in the field notes. See Section 5.5 for the specific information that must be included.

4.2.5.6 Groundwater Sampling Techniques

a. Equipment Considerations

1. Some pumps may be used for sampling groundwater. All notes and restrictions as defined in Table 4.1 and discussed in Purging and Sampling Equipment (Section 4.2.5.3) shall be followed when using pumps to collect samples.

a. NOTE: The only pump that is currently approved for use in collecting volatile samples is an all stainless steel and teflon bladder pump.

2. Other than the actual sampling device, intermediate vessels should not be used during the sample collection process. This is especially true of any compound where loss of sample is a problem (O&G, TRPH and VOCs). For all trace compounds, the sample should come in contact with as few surfaces or vessels as possible since excessive handling can result in contamination or sample loss.

3. Dedicated Sampling Equipment

a. The use of dedicated equipment is recommended since it significantly reduces the chance of cross-contamination.

b. Dedicated is defined as equipment that is to be used solely for one location for the life of that equipment (permanently mounted pump or permanently dedicated bailer). Bringing 5 bailers on site to sample 5 monitor wells does not constitute dedication UNLESS the bailers were purchased for the project, and each bailer is specifically assigned to purge and/or sample a particular well.

c. All material construction and restrictions from Table 4.1 also apply to dedicated equipment. Equipment should be purchased with the most sensitive analyte of interest in mind.

d. Cleaning/Decontamination

1. Dedicated pumps shall be cleaned prior to installation. They need not be cleaned prior to each use but should be cleaned when they are withdrawn for repair or servicing.

2. Any permanently mounted tubing need not be cleaned.

3. Any replaceable or temporary tubing shall be cleaned as specified in Section 4.1.7.

4. Equipment blanks on dedicated pumps shall be required when the tubing is cleaned or replaced and shall be collected through that portion of the tubing that is accessible.

5. Dedicated bailers, if stored in the well, must be suspended above the water column and completely decontaminated between sampling events.

a. After sampling is complete, they shall be rinsed with tap water and/or analyte-free water, wrapped to prevent contamination, and stored on- or off-site until the next sampling event.

b. The sampling equipment shall be decontaminated prior to on-site arrival UNLESS the equipment is stored on-site. In the latter case, the dedicated bailer shall be fully decontaminated prior to on-site use.

c. A precleaned equipment blank shall be collected prior to reintroducing the cleaned bailer into the water column.

b. Sampling with Bailer

1. When a bailer is used for sampling, the integrity of the sample collected is highly dependent upon the sampler's skill and familiarity with proper sampling techniques.

2. It is recommended that for a particular site only two persons perform sampling to minimize personnel handling variation.

3. Just prior to sampling, several bailer amounts of sample groundwater shall be collected to rinse the bailer.

a. Discard the water appropriately (see Waste Disposal).

b. This should not be done if the analytes of interest include Oil & Grease, TRPH, etc. (see Section 4.0.3). As stated earlier, intermediate vessels or sampling equipment are never rinsed if these compounds are to be sampled.

4. All collection activities shall be done carefully so as to not stir up any sediments.

5. The following procedure describes general bailing techniques:

a. Field personnel should wear protective gloves (see Section 4.0.2).

b. Attach a fresh length of monofilament or braided nylon line to the bailer. Alternately, a precleaned permanent lanyard may be used.

c. The bailer or lanyard must not be allowed to touch the ground during purging or sampling.

- d. Lower the bailer slowly and gently into contact with the water so that agitation of the water column is minimized.
 - e. Attempt to sample from the same depth in the well each time, preferably within or just above the screened zone of the well.
 - f. Do not allow the bailer to touch the bottom of the well so that bottom sediment is incorporated into the sample.
 - g. Retrieve the bailer smoothly. Collecting the lanyard between the thumbs of each hand seems to be the preferred method.
 - h. Discard the first few inches of water in the bailer and fill the appropriate sample bottles so that a minimum of turbulence is created to avoid aeration.
 - i. Discard the last few inches of water in the bailer.
 - j. Add preservatives (if necessary), check the pH of all pH-adjusted samples (except VOCs).
 - k. Attach and/or complete the sample container container labels, record information in field notes, place samples on wet ice (if required) and protect all samples from sun.
6. Sampling with disposable bailers, though acceptable, is not recommended as a standard procedure for environmental sampling.
- a. Disposable bailers of the appropriate construction material are available. High density polyethylene (HDPE) bailers are acceptable for all inorganic parameters (and free product thickness).
 - b. Teflon bailers are also available as a disposable for use where organics are of concern.
 - c. As the agency charged with solid waste management as well as environmental sampling and analysis, the Department cannot encourage the use of disposable equipment for all situations. In situations where expensive, permanent sampling equipment may be destroyed or damaged by sampling a concentrated waste, the use of disposable equipment is recommended.
 - d. Precleaned equipment blanks are required for disposable equipment (see Quality Control for frequency).

c. Sampling with Pumps

As a general rule, pumps shall not be used to collect samples if organics are of interest. There are two exceptions: 1) use of the peristaltic pump with a trap

(see Fig. 4.1 for specific configuration) for EXTRACTABLE organics; and 2) use of an all Teflon and stainless steel bladder pump for all organics.

1. Peristaltic Pump

a. Organics

1. Assemble the components of the pump according to the Fig. 4.1.

2. The container shall be a glass or teflon bottle. The sample container is recommended, however, if an intermediate vessel is used, it shall be decontaminated between wells per Section 4.1.4.1.

3. All equipment that contacts the groundwater BEFORE the sample container shall be of Teflon, stainless steel or glass construction, including the transport tubing to and from the sample container, the interior liner of the container cap and all fittings. UNDER NO CIRCUMSTANCES CAN A RUBBER STOPPER BE USED AS THE CAP.

4. Connect the outflow tubing from the container to the influent side of the peristaltic pump.

5. Turn the pump on and allow the container to fill approximately 1/4 full.

6. Turn the pump off, disconnect the container, rinse the bottle and discard the contents.

7. Repeat the process a second time. Note restrictions on rinsing in Section 4.0.3.

8. Turn pump on to fill the container.

9. If an intermediate container is used, distribute the sample into appropriate containers.

10. If the sample container is used, discard a small portion of the sample, to allow an air space.

11. Preserve (if required), label and complete field notes.

b. Inorganics

1. Inorganic samples may be collected from the effluent tubing, and there are few restrictions on tubing type (see Table 4.1).

2. If samples are collected from the pump, all tubing (including the tubing in the head) shall be changed between wells.

2. Bladder Pump

a. The flow rate shall be reduced after purging to a smooth, even flow.

b. When sampling for VOCs, the flow rate must be reduced to 500 ml/minute (approx. 0.1 gallon/min).

3. Other pump types

- a. Sampling for INORGANICS ONLY may be conducted with most other pump types (see Table 4.1).
- b. The flow rate during sample collection shall be a smooth even flow.
- c. All tubing and the pump shall be decontaminated between wells.

d. Sampling Wells that have Free-Phased Product

[[1. The Department does not recommend the sampling of wells with free floating product for trace contaminants. This concerns primarily petroleum related sites, but includes any chemical product (e.g. solvent) that floats on the water table. Sampling is acceptable if the information is to be used for the purpose of remedial design.

2. Sample data from such wells cannot provide useful information regarding the level of contamination. Furthermore, the Department believes that these wells may never provide legitimate data as they may have become (permanently) chemically damaged by the product being in contact with the well casing for an extended period of time.

3. The Department does reserve the right to require sampling of these wells, not for levels of trace contaminants, but for confirmation of an appropriate remediation technique. This type of sampling is performed BELOW the product layer (see 4.2.5.6.f. below).]]

e. Free Product Sampling

1. Free product is normally sampled for two reasons:
 - a. documentation for its existence (and thickness), as required in the Tanks regulations; and
 - b. determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases as weathered product may not be able to be identified.

2. Free product may be evident in a cased monitor well or an open excavation.

3. It is recommended that plastic (acrylic, clear PVC) bailers be used for sampling the monitor wells. Optionally, disposable (HDPE) bailers are acceptable. Other wide-mouth vessels may be used for sampling the excavation.

a. Monitoring well:

1. If free product (defined in 17-770 as product in excess of 0.1 inch) or product globules are present in a monitoring well, a precleaned bailer is used to collect the sample.

2. Once the bailer is withdrawn product thickness is measured to the closest 0.1 inch.

3. A portion of the product is poured into a glass vial.

4. As a concentrated waste, this sample must be wrapped to prevent breakage, isolated from other samples, iced to 4 C, and proper chain of custody information completed.

b. Excavation:

1. If free product is observed in an open excavation, the glass sample container or a precleaned intermediate vessel may be used to collect the sample.

2. A lanyard (e.g. braided nylon) is tied to the container and lowered into the excavation.

3. Care must be taken not to introduce solid material into the container as it is being lowered or retrieved.

4. If sufficient water is available, a bailer can be used.

5. Though not recommended, screened casing can be placed (or augured and placed) in the bottom of the excavation and sampled with a bailer.

6. Avoid dangerous situations, such as standing too close to the edge of an excavation, riding in the backhoe bucket, or entering a trench or excavation that may collapse.

7. All applicable OSHA regulations should be followed.

4. Equipment which is dedicated to sampling free product does not need to be cleaned according to the standard, full decontamination protocols. Acrylic or PVC bailers that are never used for trace contaminant sampling may be cleaned as listed below. It is recommended that all cleaning be done in the lab, office, or base of operations and not in the field.

a. disassemble bailers and intermediate vessels and soak in hot, soapy tap water using a brush to clean away all particulates and greasy films,

b. rinse with hot tap water,

c. thoroughly rinse with DI water

d. An optional acid rinse may be used to strip the equipment of any hard to clean residues.

e. The solvent rinse is not mandatory since this equipment is not used for contaminant sampling, other than the product itself. It is not recommended on clear acrylic.

f. Sampling Below Product

1. This type of depth-specific sampling is performed only at the request of DER or its designee. An attempt is made to sample the dissolved constituents in the water column below the product layer.

2. These data provides information that helps define adequate groundwater treatment. Without these data, incorrect (and sometimes unnecessarily expensive) remediation techniques may be designed for a situation where they are not required.

3. There are some substantial logistical problems involved with sending a sampler through free product to sample the groundwater below. Although there are some products designed specifically for this type of sampling, they are expensive and the results may not be commensurate with their cost. The use of "self-engineered" equipment or coverings may be the best option.

4. These data are only to be used for qualitative use and will aid in deciding on an appropriate remediation technique.

5. Wrapping bailers and tubing in plastic seems to be the most popular technique in getting past the product layer.

6. Though not recommended, some have wrapped submersible pumps in several layers of plastic and retrieved each layer by a separate lanyard.

7. One suggestion would be to use a rigid piece of stainless steel tubing wrapped in plastic.

a. Once the covered tubing is past the layer, pull up on the plastic, piercing the plastic and exposing the (somewhat) clean tubing inlet.

b. Introduction of the wrapped hose must be done slowly so as to not entrain any more product into the dissolved layer located below.

c. Also, this must be done with a peristaltic pump or a vacuum pump linked to a trap bottle. To use this setup, the water table must be no deeper than 15-20 feet, realizing that actual sampling may be occurring several feet below the product layer.

g. Sampling Dissolved Metals

1. In order to collect a "representative" sample for the purpose of monitoring compliance with groundwater standards for metals, it may be necessary to field filter a sample prior to preservation.

2. In situations where the static level in the well allows use of a peristaltic pump, the groundwater sample shall be pumped directly from the well through an in-line filter.

- a. A disposable, high capacity, .45 um filter is an acceptable filter for most applications. See Fig. 4.2 and Table 4.1 for allowable equipment setups.
 - b. In field use, the filter must first be flushed with 30 - 50 mls of deionized water or an inert gas to remove atmospheric oxygen.
 - c. The filter must be inserted on the high pressure side (i.e. on the delivery side) of the peristaltic pump. VACUUM FILTRATION IS NOT ACCEPTABLE.
 - d. The sample delivery tube must be long enough (greater than 2 feet) such that back-diffusion of oxygen to the filter is negligible.
 - e. New or precleaned silastic tubing shall be installed in the pump at each monitor well.
3. In situations where the static water level in the well is too deep for a peristaltic pump to be used directly, there are several alternatives:
- a. Groundwater may be sampled with an appropriately constructed bailer. The intake tube of the peristaltic pump is inserted into the full bailer and water pumped through a filter as described above.
 - b. Any submersible pump of appropriate construction for which the flow rate can be adjusted may be used for water levels below 20'-25'.
 - c. Pressurized HDPE and Teflon bailers may also be used.
 - d. See the specific section concerning field filtration in Table 4.1 for all acceptable alternatives.
4. It is important that this operation is carried out as rapidly as possible and in such a way that sample agitation and exposure to atmospheric oxygen is minimized. It is for this reason that pouring the sample into any intermediate vessel for subsequent filtration IS NOT allowed. This includes barrel or syringe filters. Once the sample is collected into a sample container, preservation and pH checks should be completed.

4.2.6 Wells with In-Place Plumbing

Wells with in-place plumbing are generally encountered at wellfields, industrial facilities and private residences. See separate discussions below on sampling potable water wells.

4.2.6.1 Purging

- a. The volume to be purged depends on several factors: the depth and diameter of the well, whether the pumps are running continuously or intermittently, how close to the source the sample can be collected, and the presence of any

storage/pressure tanks between the sampling point and the pump.

b. If storage/pressure tanks are present, an adequate volume must be purged to totally exchange the volume of water in the tank (EPA, 1986).

c. Continuously Running Pumps

1. If the pump runs continuously and the sample can be collected prior to a storage/pressure tank, no purging is required, other than opening a valve and allowing it to flush at maximum velocity for at least 15 minutes.

2. If the pump runs continuously, and a storage/pressure tank is located ahead of the sample location, the purge must include the entire storage volume to ensure that a sample representative of the groundwater will be collected.

d. Intermittently Running Pumps

1. If the pump runs intermittently it is necessary to determine the volume to be purged, including storage pressure tanks that are located ahead of the sampling location.

2. The pump should then run continuously until the required volume has been purged.

3. When the well depth or diameter is unknown (as is frequently the case with in-place plumbing) purging should be carried out by pumping the well for 15 minutes and until the pH, specific conductance and temperature stabilize.

a. In practice, stable sample chemistry is indicated when the purging parameter values remain within 5% over two successive samples taken at least 5 minutes apart.

4.2.6.2 Sampling

All samples must be collected from the closest spigot to the well head, with all screens or aerators removed, and with the flow rate reduced to no more than 500 ml/min.

4.2.7 Potable Well Sampling

The following procedures describe generalized drinking water sampling from private potable wells. If the samples are collected for compliance with the drinking water regulations (Chapters 17-524, 17-550 or 17-555, F.A.C.), the samples must be analyzed by a laboratory with Drinking Water Certification. If the samples are being analyzed in response to other programs (contamination assessment, consent order, etc.), the laboratory shall meet the requirements of the specified Category.

4.2.7.1 General Concerns

- a. Appropriate containers and preservatives must be selected prior to sampling.
 - 1. Containers and preservatives shall comply with Tables 4.2, 4.3, 4.4 and 4.5.
 - 2. Containers and preservatives may be obtained from a laboratory with appropriate credentials (see discussion above).
 - 3. It is recommended that the laboratory add the appropriate preservative to the container.
- b. The laboratory may include special handling instructions with the sample containers. These must be read carefully and must comply with the generalized instructions listed below.

4.2.7.2 Sampling Drinking Water Wells

- a. As a general rule, purging and sample should be from a spigot closest to the well head.
 - 1. If possible, the spigot should be before the holding tank and filters. If this is not possible, the contents of the holding tank must also be purged.
 - 2. Remove all aerators and filters (if possible).
- b. Depending on the running schedule of the well and the placement of the pressure tank, purge the system as described in Section 4.2.6.1.
- c. If the capacity of the pressure tank is not known, purge for at least 15 - 20 minutes at maximum velocity.
- d. Reduce flow to approximately 500 ml/minute (a 1/8" stream).
- e. Sample Containers with no preservatives:
 - 1. Remove the screw cap from the bottle. Do not touch the interior of the cap or the container with hand or the spigot.
 - 2. Fill approximately 1/4 full, rinse the interior of the container and discard the water.
 - 3. DO NOT RINSE CONTAINERS IF collecting samples for oil and grease, total recoverable hydrocarbons, volatile organics (including trihalomethanes) or microbiologicals.
 - 4. Tilt the container so that flow falls onto the interior surface. DO NOT AGITATE OR SHAKE CONTAINER WHILE FILLING.

5. Fill the bottle to almost to capacity (if collecting VOC or trihalomethane samples, see 4.2.7.2.i below).

6. Replace the screw cap securely on the bottle.

f. Sample containers with preservatives.

1. Follow the same protocol outlined above, deleting the rinse.

2. Since some of the preservatives may react with the sample water, hold the open end of the container away from you while filling.

3. After replacing the cap, gently tip the container several times to mix the preservatives.

g. Affix a sample label and seal (if required), and complete the chain-of-custody form.

h. Place the sample bottle in a plastic sample bag and cool to 4 C on wet ice.

i. Special Sampling Protocols

The special precautions for the types of samples discussed in Section 4.2.2 shall be followed.

4.2.7.3 Sampling Drinking Water Sources for Lead and Copper

a. Selection of the sampling point is dependent on whether the sample is being taken to verify compliance with the Drinking Water Regulations. If so, the sample must be collected from a COLD WATER tap in either the kitchen or bathroom.

b. Samples must be collected after the water HAS NOT been used for at least SIX HOURS.

c. DO NOT FLUSH OR PURGE THE SYSTEM.

d. Collect the first flush into the sample container for trace metals. DO NOT RINSE SAMPLE CONTAINER.

e. Tilt the container so that the initial flow falls onto the interior surface. DO NOT AGITATE.

f. If the container was prepreserved, hold the open end of the container away from you while filling.

g. Add preservatives (if needed).

h. Replace screw cap and gently tip the container several times to mix the preservatives.

i. Affix a sample label and seal (if required), and complete the chain-of-custody form.

j. Place the sample bottle in a plastic sample bag.

4.2.8 Drinking Water Supply System Sampling

The following protocols shall be followed:

1. When sampling for drinking water compliance, the sampling spigot is normally designated by permit or municipal authorities.

The location may be near the supply line or may be an outside spigot on a private residence.

2. Procedures to sample drinking water directly from the supply system is the same as above, except for treatment of residual chlorine.

- a. Lines shall be flushed for 2 to 5 minutes before collecting any samples.
- b. Reduce the flow rate to less than 500 ml/min (1/8" stream) before collecting samples.

3. In many instances, the water supply to residences may be treated with chlorine which may cause interference with certain types of analyses (ex: VOC; Semi-Volatiles and some bacteriological samples). Residual chlorine must be treated with the addition of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$).

4. Utilizing chemical kits (such as HACH), test the water in a separate container for residual chlorine. If residual chlorine is present, collect the sample in the appropriate sample container(s) using the required preservatives.

- a. Immediately upon sample collection add 0.008% $\text{Na}_2\text{S}_2\text{O}_3$ or 100 mg of $\text{Na}_2\text{S}_2\text{O}_3$ per 1 liter of sample water directly into the sample container.

- b. After replacing the cap, tip the container several times to mix the preservative.

5. Affix a sample label, seal and transport on wet ice.

6. Lead and copper shall be sampled according to protocols outlined in 4.2.7.3.

4.2.9 Temporary Well Points

Temporary well points include those drilled with augers as well as those pushed with "direct push" or DPT devices. These types of wells are not permanently installed.

4.2.9.1 Use

a. Temporary well points may be used for PRELIMINARY INVESTIGATIONS and as a SCREENING TOOL.

[[b. For formal site work (not preliminary or PCAP), temporary well points may only be allowed under emergency situations. These are:

1. DOT right-of-ways,
2. private property where a permanent well cannot be placed, or
3. inside or up against a structure.]]

c. DER will determine whether temporary well points are warranted.

d. If these wells are used to provide formal data, these restrictions apply:

1. Use precleaned equipment as described in Table 4.1;
2. Well must be purged of 3-5 well volumes (or dry);
3. Sampling with a peristaltic pump

- a. Extractable organics shall be collected via an all-Teflon and -glass organic trap configuration (see Figure 2.1);
 - b. VOCs shall not be collected through a pump, but the Teflon pump tubing is allowed to fill via ambient pressure, capped with stopper or gloved finger, carefully withdrawn from the well, and drained into appropriate vials.
 - c. Refer to protocols listed in 4.2.5.5 and 4.2.5.6 for specific information on sampling and configuration.
4. Sampling with bailers
- a. In some cases, sampling may be accomplished with a 3/4" bailer.
 - b. All equipment construction restrictions shall be followed.
 - c. Refer to bailer sampling protocols in section 4.2.6.5.

4.2.10 Airstripper and Remedial Treatment System Sampling

- a. Collect effluent samples from airstripper units in a similar manner to those described for Drinking Water Supply Systems (Section 4.2.8).
- b. Remove any tubing from the sampling port and flush for one to two minutes.
- c. Reduce flow rate to less than 500 ml/min. and begin sample collection.

4.2.11 Bioassay Sampling

When collecting samples for bioassays, the sampling protocols outlined in Section 4.2.3 (Surface Water) and 4.2.4 (Wastewater) shall be followed.

The holding time for bioassay samples is 72 hours.

4.3 SOLID MATRIX SAMPLING PROCEDURES

This section is concerned with grab and (areal or depth) composite samples from solid matrix (soil and sediment). Since similar procedures and equipment exist for soils and sediments, a general description of sample handling will be discussed. EPA and the Department define sediments as the solid matrix material that is located beneath surface waters. Although terrestrial soil is "sedimentary" material, these matrices are considered distinct.

4.3.1 General Concerns

4.3.1.1 Sampling equipment shall be selected based on the type of sample to be collected and the parameters of interest. See Table 4.1 for specific requirements and Section 4.2.5.2 of the Groundwater section for a discussion on material.

4.3.1.2 All equipment shall be decontaminated according to specified protocols in Section 4.1.

4.3.1.3 All general sampling concerns outlined in Section 4.0 shall be followed.

4.3.1.4 Field activities shall be documented in accordance with requirements in Section 5.

4.3.1.5 Sample container and holding time requirements listed in Section 4.4.2.3 shall be followed. The sample containers shall be cleaned or obtained according to protocols listed in Section 4.4.1.1.

4.3.2 Sample Handling Protocols after Sample Acquisition

General sample handling will fall into 3 main categories; surface, shallow subsurface, and deep subsurface. Each of the three categories will be discussed in general. Once the sample is acquired, the handling procedures are very similar and are described below.

1. Select the appropriate precleaned sampling device and procure the sample from the desired depth. If using liners to transport the sample to the lab, see 4.3.2.10 below.
2. Select the required sample container for the parameter group.
3. Split spoons and Shelby Tubes
 - a. Breakdown the sampler (split spoon, Shelby tube). This should be done with the appropriate tools.

b. At this time, any portion of the sample that has been disturbed shall be identified, removed with a stainless steel spatula and discarded.

c. Slice the sample using a clean, decontaminated stainless steel spatula from the center portion of the corer, split spoon or bucket auger head.

d. For VOC analyses, immediately transfer the sliced portion to a suitable container (the container must be equipped with a teflon-lined septum seal).

1. Carefully fill the vial (or wide-mouth container) with sample.

2. Tamp the sample into the vial with a SS, glass, or Teflon rod to reduce headspace.

3. Add sample and tamp down until no headspace exists.

4. After cleaning exterior and rim of vial as described below, cap container (with teflon side facing sample) tightly, label and place on wet ice immediately.

e. For other analyses, slice sufficient amount of sample from the center portion of the sampling device and transfer it to a tray of appropriate construction (note restrictions on use in Table 4.1).

4. Bucket Auger, Dredge or Corer

a. Remove the sample from the sampler (bucket auger, dredge, corer) with appropriate (stainless steel, teflon, etc.) tools and place in a stainless steel, glass or aluminum foil-lined tray (note restrictions on use in Table 4.1).

b. Remove any portion of the sample that has been disturbed with a stainless steel spatula and discarded.

c. If VOCs are required, fill an appropriate container with aliquots that have been taken from selected areas of the entire sample. Proceed as described in 4.3.2.3.d above.

5. Sample Mixing

a. VOCs shall be collected as discussed above before the sample is mixed.

b. The sample in the tray shall be homogenized thoroughly:

1. Appropriate tools shall be used to mix the sample.

2. Homogenize by alternately mixing, dividing, and remixing the sample.

c. After thorough mixing, transfer the sample to the appropriate sample container(s) leaving minimal headspace.

6. Clean the outside of the sample container to remove excess soil.

7. The container rim should also be cleaned of soil and sand particles so that the lid can be sealed. An improperly sealed

container may allow cross contamination from ice melt or petroleum fumes.

8. Affix sample label, seal (if applicable), and complete the chain-of-custody forms.

9. Place the sample containers in a clean, plastic sample bag and preserve by placing in wet ice.

10. Liners

- a. If properly used, liners may be inserted into the sampler and used as the actual sample container.
- b. Be aware that SW-846 has mandated that all solid samples must be transported in containers that have screw tops. This also means that all container and lid requirements are still in effect.

1. For inorganic samples, ends of the liner must be covered with polyethylene, Teflon, or aluminum foil sheeting. The sheeting should be secured by placing an end cap over the sheeting.

2. For organic samples, the sheeting must be Teflon or aluminum foil.

3. With any sample containerized this way, specific instructions must be sent with the sample so that the laboratory will know how to handle the sample. All non-volatile samples must be homogenized by the laboratory prior to analyses. Also, any disturbed portions of the sample should be discarded prior to mixing.

4.3.3 Composite Soil Samples

The following is not a complete discussion regarding development of a sample compositing scheme nor all available sampling protocols. When a large site area is to be investigated for contamination, it is sometimes advantageous to composite soil or sediment samples and thus minimize the number of samples to be analyzed.

1. Sample aliquots (of identical size) to be composited shall be placed in a tray of suitable materials (see Table 4.1) and thoroughly mixed with a cleaned spoon, spoonula or spatula of suitable materials (see Table 4.1). The sample shall be thoroughly blended by mixing, and dividing into sections. Each section shall then be mixed separately. Recombine all mixed sections and mix thoroughly. Repeat sectioning and mixing process to ensure proper homogenization.

2. The origin and size of each (sub)sample or aliquot that is used to make the composite shall be documented in the field notebook along with the other important sampling details.

- a. Although the size of these subsamples is important and should be documented, it is critical that these subsamples be of equivalent size, so that the composite sample is not biased by unequal aliquoting.
 - b. There is no level of accuracy here; it is dependent upon the size of the aliquots.
 - c. Aliquoting should be done in a systematic manner.
3. Samples for VOCs shall not be composited or mixed unless the DER programs or permits require compositing. This procedure should only be undertaken if mandated by a formal DER document (permit, Consent Order, etc.) and shall follow these protocols:
- a. Composite samples for VOCs shall not be mixed as described above.
 - b. Equal portions of all samples to be included in the composite shall be placed in the VOC container (see 4.3.2.3.d above).
 - c. The laboratory shall be informed that the sample is a composite and must be mixed before analysis.
4. Clean the outside of the sample container to remove excess soil, affix label, seal (if required), and complete the laboratory transmittal forms.

4.3.4 Soil Sampling

[[4.3.4.1 Site Selection

- a. Soil sampling locations should be selected such that a representative portion of the soils are collected with minimal disturbance. Locations where natural vegetation is stressed or dead and/or areas that have surficial soil staining may be indicative of improper waste disposal practices.
- b. An upgradient, undisturbed location should also be selected for obtaining background and/or quality control samples. Be aware that differences in soil types may affect these background samples (e.g. sands vs. clays).]]

4.3.4.2 Surface Soil Sampling - ground surface to 6 inches below ground surface

- a. Leaves, grass and surface debris shall be removed from the area to be sampled using a clean stainless steel spoon or shovel.
- b. Surface soil samples can then be collected using a precleaned stainless steel scoop or spoon.

4.3.4.3 Shallow Subsurface Soil Sampling

- a. Shallow subsurface samples may be collected by digging a hole or trench to the required depth with a stainless steel shovel.

b. Some situations may require a trench or pit to be dug with a backhoe. Depending upon the equipment available at the site or the soil type to be penetrated, this option is acceptable. [[Please note that any OSHA requirements for in-trench sampling should be followed.]]

1. In these situations, the trench is first dug to the appropriate depth and then the sample is exposed by using one precleaned spoon, spatula, or equivalent to clean away the soil that came in contact with the backhoe bucket and a second precleaned spoon to actually collect the sample.

c. Alternatively, shallow subsurface soil samples may be collected with 2-4 inch stainless steel bucket auger which would minimize the soil to be removed in order to reach the desired depth. Using this method, a sampling depth of up to 15 feet may be obtained.

1. The bucket auger consists of a stainless steel cylinder with flush welded stainless steel cutting edges. The cutting edges are hardened surfaces, heat treated and sharpened.

2. A soil sample is obtained by pushing and rotating the auger into the soil until the bucket is filled.

3. The sample can be removed (with some difficulty) from the bucket by pushing or scraping with an appropriate precleaned stainless steel tool.

4. This auger method is useful for obtaining large samples of unconsolidated sediment.

5. The device is supplied with 3 foot extension rods.

6. Addition of a sleeve may allow an undisturbed soil sample to be obtained.

- a. The device consists of a standard auger head with a removable non-contaminating sleeve which is inserted into the auger barrel.

- b. Either a clear butyl acrylate (CAB) plastic sleeve (for inorganic samples) or stainless steel (for organic samples) may be utilized.

- c. The soil sample is obtained in the normal manner by pushing and rotating the auger into the soil. In this case it is the sleeve which fills with soil. After auger retrieval, the sleeve, which is readily removed from the auger, is capped (see Section 4.3.2.10 above).

7. If the auger hole is prone to collapse, due to low cohesion in some soils, a temporary rigid PVC casing should be inserted into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced.

d. Upon sample collection, the temporary casing (if used) must be removed and the hole filled with the excavated soil.

[[e. If a confining layer has been breached during sampling, the hole shall be grouted to land surface with Type-1 Portland cement. NOTE: this requirement may be different throughout Florida. Contact the local Water Management District office for local requirements.]]

4.3.4.5 Deeper Subsurface Soil Sampling

a. A drill rig is normally required if soil samples are taken from boreholes greater than 15 feet BLS (below land surface). There are a number of sampling devices used in conjunction with the drill rig for retrieving the samples; Shelby tubes, split spoon samplers and standard core barrels.

b. Shelby Tube Sampler

1. The Shelby tube sampler is used to sample unconsolidated soils and consists of a stainless steel tube approximately 30 inches long and 2 inches, or larger, in diameter.

2. One end of the tube has edges beveled into a cutting edge. The other end can be mounted to an adapter which allows attachment to the drill rig assembly.

3. After drilling to the required depth with an auger or rotary drill bit, a soil sample is obtained through the auger or directly in the borehole.

4. The Shelby tube is pushed into the soil using the drill rig's hydraulic ram or manually with a sledge hammer.

5. When the tube is retrieved, the soil sample taken from the center and away from the sides, can be transferred into the appropriate container (for VOCs) and/or mixed using a stainless steel spoon handle or spatula when other parameters are of interest.

c. Split Spoon Sampler

1. A split spoon sampler, useful for sampling unconsolidated soils, consists of two carbon steel half cylinders (spoons) that fit together to form a tube approximately 2 feet in length and 2 inches in diameter.

2. The cylindrical arrangement is maintained by a retaining head and bit rings that screw on at either end of the split spoon.

3. The bit ring has beveled edges to facilitate sampling as the split spoon is forced into the ground.

4. As with the Shelby tube, either the weight of the drill stem and rods or a mechanical hammer is used to advance the sampler.

5. A catcher device is inserted in the head ring to prevent loss of unconsolidated sample during recovery.

6. After retrieving the split spoons, the soils can be withdrawn by unscrewing the bit and head rings and splitting the barrel.

7. The top 2 to 3 inches of the sample will be normally disturbed and should be discarded.

8. A cleaned stainless steel spatula is used to collect a subsample for VOCs and/or transfer the contents into an appropriate tray for mixing and containerizing.

d. Standard Core Barrel

1. A standard core barrel is utilized when consolidated samples (such as limestone or dolomite) are to be sampled.

2. The core barrel is carbon steel cylinder approximately 3 feet long and 2 inches in diameter.

3. The barrel has a removable head ring with small embedded diamonds which allow the device to cut through rock or consolidated soils as the drill rods are rotated.

4. The sample core can be retrieved by unscrewing the head ring and sliding the sample into the container.

4.3.5 Sediment Sampling

4.3.5.1 General Overview

a. Sediment samples are taken from material underlying streams/rivers, lagoons, ponds/lakes, and estuaries.

b. The actual sampling location is dependent upon project scope.

c. Sediment samples may be taken as an adjunct to surface water samples.

d. They may be taken as a compositing series to define water or sediment quality in a system.

e. They may be taken above and below an outfall to document degradation.

f. Similarly, if stressed shore vegetation or visible surface water contamination is evident, sediment samples may be taken.

g. Decisions for sample location will not be discussed in this document.

h. All surface water samples shall be taken prior to any sediment samples (see 4.2.3.2).

4.3.5.2 Sample Collection Protocols

a. Sediment samples are taken via three groups of equipment: scoops; corers and dredges.

b. Soil sampling equipment is generally not applicable to sediments because of low cohesion of sample.

c. Sample location (edge or middle of lagoon), depth of water and sediment, sediment grain size (fineness), water velocity, and analytes of interest all must be considered when choosing equipment.

d. Stainless steel equipment shall be used if trace contaminants are to be sampled.

e. Dredges must be used for hard or rocky substrates. They are heavy enough to use in high velocity streams.

f. Coring device may be used for softer substrates. Coring devices must be used in soft substrate if the fine particles are to be included. Coring devices should be used in quiescent waters.

g. Scoops

1. Scooping is generally most useful around the margin or shore of the water body.

2. Stainless steel spoons or grain scoops work very well. The scoops can be attached to an extendible pole for obtaining samples several feet from shore or boat.

3. The sampler may also wade into the water body to obtain a scooped sample.

4. The sampler must stand facing the direction of flow and approach the location from the downstream direction.

5. Precautions must be taken not to disturb the bottom prior to scooping.

6. The sample shall be scooped in the upstream direction of flow.

h. Corers

1. Coring devices can be easily fabricated from many materials. Although stainless steel, glass or teflon must be used for sampling trace organics, other inexpensive material (PVC, carbon steel, etc.) may be used for demands, nutrients, metals as appropriate.

2. Some corers are simple "push tubes", whereas other more sophisticated models may be finned, gravity driven devices.

3. Not only are they useful in sampling fine grain sediments, they can also present or preserve the historical layering of sediments.

4. Upon descent, water displacement is minimal, which also minimizes the shock wave produced by other equipment (dredges).

5. The corer is the equipment of choice for fine sediments in static waters, especially trace organics and metals.

6. Corer diameter, grain size, and sample consistency will determine if the sample will remain in the corer upon withdrawal.

7. Sample washout can be a problem and there are several ways to reduce or prevent it.
 - a. The leading edge of the corer can be fitted with a nosepiece or core catcher which physically keeps the sample from slipping back out the corer.
 1. The core catcher material must also be compatible with analytes of interest.
 - b. A second option is fitting the top or back end with a check valve which first creates negative pressure on the back of the sample as it is being pulled from the substrate and second, prevents surface water from washing out the top portion of the sample.
 8. The corer shall be rotated as it is pushed in.
 - a. Rotation should be around its axis, not rocked back and forth.
 - b. Rotation improves penetration and prevents compaction of the sample as it is pushed to the full length of the corer.
 9. Upon withdrawal from the water surface, a cap shall be placed on the bottom to prevent the sample from sliding out.
 10. The core should then be extruded out into a pan or tray and sample processed as described in Section 4.3.2 above.
 11. Corers can also be fitted with liners. This is advantageous if a complete core is desired that has not been in contact with the atmosphere. It is also advantageous if the coring device is not constructed of the proper material (e.g. PVC) and one of the analytes requires a sampler of inert construction (glass, SS, or Teflon).
- i. Dredges
1. The three main types are the Peterson, Eckman, and Ponar.
 2. The Peterson and Ponar dredges are suitable for hard or rocky substrates.
 - a. The Peterson and Ponar are virtually the same, except the Ponar has been adapted with a top screen and side plates to prevent sample loss upon ascent. For this reason, the Ponar is the dredge of choice for rocky substrates. These dredges are heavy enough to use in streams with fast currents.
 3. The Eckman is designed for softer substrates of sand, silt, or mud.
 - a. The Eckman is too light to use in fast currents.
 4. Follow the manufacturer's suggestions for setting and operating the weighted messenger devices.

4.3.6 Fish-Tissue Sampling

4.3.6.1 Decontamination

a. It is important to properly decontaminate sampling equipment prior to going into the field and between individual fish, as heavy metals and organic compounds are usually the analytes of interest in fish tissues and fish slime contains these analytes. Therefore, all equipment used for tissue sampling shall be decontaminated using the following procedure:

1. Rinse equipment with tap water.
2. Wash with Liquinox or Acatonox (or a comparable detergent). A brush may be required to remove scales and other tissue remnants.
3. Rinse with deionized water.
4. Rinse with isopropanol.
5. Use analyte-free water for a final rinse.
6. Decontaminated equipment should be wrapped in aluminum foil or untreated butcher paper to protect it from contamination if it will be stored or transported prior to use.

NOTE: If analyte-free water is unavailable, allow isopropanol rinsed equipment to dry thoroughly before use.

4.3.6.2 Sample Collection/Sample Preparation

- a. Fish shall be captured using the means appropriate for the situation (electroshock, seine net, hook and line, etc.) and immediately placed on wet ice in coolers.
- b. Balances, cleaned by the method above and rezeroed after weighing each fish, should be used to weigh the fish as soon as possible after capture.
- c. NOTE: if the fish are to be used for the analysis of dioxins by Method 8090, the following procedures for filleting SHALL NOT be followed. WHOLE fish shall be frozen and transported to the laboratory for processing.
- d. The filleting procedure should take place on a stable surface. The surface shall be constructed of stainless steel or other suitable materials (see table 4.1) which can be decontaminated initially and after each fish is prepared.
- e. Using a clean stainless steel fillet knife with either a wooden or stainless steel handle, the individual fish should be filleted carefully, so as not to puncture any visceral organs.
- f. The sampler should wear disposable gloves (see Section 4.0.2) which must be changed between fish.
- g. Fillets shall be scaled, but not skinned, as both skin and muscle tissue are considered edible portions of the animal. (This is especially important if organic

analytes are being sampled, as these compounds are fat soluble and a layer of fat lies directly below the skin.)

h. Duplicate samples shall be collected from the same regions of opposite fillets, labeled, stored and analyzed as duplicates.

i. Samples may be of any size and may include the whole fillet, but must be taken through the entire width of the fillet if smaller.

j. All samples taken shall be wrapped in clean aluminum foil, labeled and placed in a plastic bag.

k. At a minimum, samples shall be preserved by keeping them on wet ice while in the field for no more than 24 hours and shall be frozen upon return to the laboratory. It is important to guarantee that the moisture content does not drop through repeated freeze-thaw cycles.

l. Optimal preservation of fish tissue samples should include lyophilization (freeze-drying) combined with determination and recording of the sample's moisture content (weight loss) after freeze-drying. The latter technique is often beyond the scope of many laboratories and most consultants.

4.3.6.3 References:

1. Mid-America Fish Contaminants Group. August 1989, Fish Sampling Guidelines.
2. DER Water Quality Monitoring and Quality Assurance Section, Bureau of Water Analysis, Tallahassee, FL. July 1984 Draft, Interim Method For The Sampling and Analysis Of Metals In Fish Tissue With Emphasis On Mercury.
3. U.S. E.P.A., Environmental Monitoring and Support Laboratory, Cincinnati, OH. October 1980, Interim Methods For The Sampling And Analysis Of Priority Pollutants In Sediments and Fish Tissues.

4.3.7 Shellfish Sampling

This protocols pertain only to the collection of whole organisms which will be processed by the laboratory. This means that the shellfish must be collected with shells INTACT.

4.3.7.1 Sample Collection

- a. Shellfish may be brought to the surface using divers, clam rakes, tongs or any other suitable means.
- b. Unless specific species are required, the sample should include samples of all species.
- c. Select only live organisms. This means that the shells are tightly closed and cannot be manually pried open.
- d. The laboratory must be consulted for the number of shell fish to submit. Since many shells will open in

transit, collect at least twice as many specimens as the laboratory required.

4.3.7.2 Sample Transport and Preservation

- a. Wrap all specimens from one location securely in aluminum foil.
- b. Place in a sealable plastic bag, seal bag, identify contents with a field ID number and place immediately on ice.
- c. Complete documentation which must include:
 1. Description of sampling location
 2. Depth that specimens were found
 3. Species identification (if possible)
 4. Number of different types of specimens (if applicable)
 5. Collection time
 6. Collection equipment and method
- d. Samples must be transported to the laboratory within 24 hours of collection. They may be frozen for transport, but must be frozen immediately upon receipt in the laboratory.
NOTE: If samples are frozen for transport, sufficient ice and/or dry ice must be packed to that they remain frozen during transit.

4.3.8 Residual Sampling

Residuals matrix is defined as domestic waste sludge residuals. All sampling must be conducted by following EPA's POTW Sludge Sampling and Analysis Guidance Document, 1989.

4.3.9 Hazardous Waste Sampling

Hazardous Waste Sampling shall follow the protocols outlined in the following sections of the EPA Region IV "Standard Operating Procedures and Quality assurance Manual" (February 1991):

- a. Waste: Pits, Ponds and Lagoons - Section 4.12.3, pp. 3 through 5.
- b. Waste: Open and Closed Container Sampling - Section 4.12.4, pp. 5 through 8.
- c. Waste: Waste Piles and Landfills - Section 4.12.5, pp. 8 through 9.

4.3.10 Sampling Protocols for Macroinvertebrate Identification

Sample collection macroinvertebrate organisms shall follow protocols specified in these documents:

a. "Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Waters", ORD, Washington, D.C., November 1990;

b. Standard Methods for the Examination of Water and Wastewater, Part 10500, 17th Edition, APHA, 1989.

4.4 SAMPLE HANDLING

4.4.1 Sample Containers

4.4.1.1 Obtaining Clean Containers

Sample containers shall be cleaned or obtained by one of three protocols:

- a. Purchased from commercial vendors as precleaned containers. The cleaning grades must meet EPA analyte specific requirements. All records for these containers (lot numbers, certification statements, date of receipt, etc.) and their intended uses must be documented; or
- b. Obtained from a subcontracted laboratory with an approved sample cleaning and handling protocols in their Comprehensive QA Plan. The contractor must verify that the laboratory has outlined sample container cleaning protocols in its approved DER CompQAP; or
- c. Cleaned and maintained by the organization following all analyte specific container cleaning procedures as follows:

4.4.1.2 Container Cleaning Procedures

Analysis/Parameter	Cleaning Protocols (in order specified)
Extractable Organics (GC, HPLC, GC/MS and Total Phenols)	1, 2, 4, 6, (5 and 7 optional), 12
Purgeable Organics (VOCs) (GC, GC/MS, TOX)	1, 2, 4, (6 optional, methanol only), 7
Metals (Including Cr and Hg)	1, 2, 3, 4, 8, 12
Inorganics (Including Cyanide, Alkalinity, Acidity, Residues, BOD, Color, Surfactants, COD, TOC, Chloride, Turbidity, Sulfate, Bromide, Sulfide, Fluoride, Nutrients and Radionuclides)	1, 2, 3*, 4, 8, 9, 12 (* For nutrients, nitric acid should be replaced by hydrochloric acid, or hydrochloric acid may be used after the nitric acid rinse)
Oil & Grease (and TRPH)	1, 2, 3, 4, (5, 6, 7 optional), 12
Bacteriologicals	1, 2, 8, 10
Toxicity Tests (Bioassays)	1, 2, 11, 2, 4, 6 (10 optional)

- NOTES: a) New container cleaning procedures may skip steps 1 and 2.
b) This sheet does not represent all possible cleaning procedures, and deviations may be accepted on a case by case basis.

Cleaning Procedures:

1. Wash with hot tap water and a brush using a suitable laboratory-grade detergent.
 - Organics- Liquinox, Alconox or equivalents
 - Inorganic anions- Liquinox or equivalent
 - Inorganic cations- Liquinox, Acationox, Micro or equivalents
 - Bacteriologicals- must pass an inhibitory residue test.
2. Rinse thoroughly with hot tap water.
3. Rinse with 1:1 nitric acid solution.
4. Rinse thoroughly with deionized water.
5. Rinse thoroughly with pesticide-grade methylene chloride.
6. Rinse thoroughly with pesticide grade acetone or methanol (acetone only for Bioassays).
7. Oven dry at 103 C to 125 C for at least 1 hour.*
* VOC vials and containers should remain in the oven in a contaminant-free environment until needed. They should be capped in a contaminant-free environment just prior to dispatch to the field or to field sampling consultants.
8. Invert and air-dry in contaminant-free environment.
9. Container is rinsed with sample unless container already contains preservative.
10. Autoclave containers (the tops of which are covered with aluminum foil and autoclave indicator tape is applied over the top of the container).
11. Rinse with 10% HCl followed by a sodium bicarbonate solution.
12. Cap tightly and store in a contaminant-free environment until use.

4.4.1.3 Documentation

- a. Organizations that order or clean containers shall maintain records of packing slips and lot numbers (if ordered) and/or records of cleaning protocols for container lots.
- b. Cleaning records shall at a minimum record the following:
 1. Cleaning procedure;
 2. Lot numbers of reagent solvents and acids;
 3. Date of cleaning;
 4. Initials of person who cleaned containers;
 5. Lot number (date of cleaning may be used);

6. If performed, the results of quality control tests that were run on lot numbers; and

7. Any additional cleaning or problems that were encountered with a specific lot.

c. Records shall be maintained that link lot numbers (either vendor or internal) to projects and/or clients.

4.4.2 Sample Preservation and Holding Times

4.4.2.1 General Considerations

a. Proper sample preservation is the responsibility of the sampling team, NOT the lab providing sample containers.

b. It is the responsibility of the field team to assure that all samples are appropriately preserved.

c. "IMMEDIATELY" is defined as within 15 minutes. This pertains to preservation as well as filtration immediately followed by preservation (i.e. dissolved metals, orthophosphate, etc.).

4.4.2.2 Sample Preservation

a. Sample preservation shall be accomplished by obtaining prepreserved bottles from an acceptable source or actually adding preservative to the sample in the field:

1. Sample containers obtained from the subcontracted laboratory prepreserved.

a. It is the responsibility of the organization ACCEPTING these containers to make sure that they are provided by a lab that has a DER-approved CompQAP.

b. The laboratory shall supply additional same-source preservatives in suitable containers.

2. Sample containers preserved in the field after sample addition. These preservation protocols shall be followed:

a. Preservatives shall be reagent grade or of a higher grade. Unless supported by equipment blanks, the acid for metals shall be suitable for trace metals analysis

b. Fresh preservatives shall be obtained from parent stocks prior to each sampling event. Any remaining preservatives that are not in sealed ampoules SHALL NOT be returned to stock, but must be appropriately discarded.

c. Preservatives shall be transported to the field in plastic or teflon containers unless sealed by the manufacturer in glass ampoules.

d. Preservatives shall be added with disposable pipettes or premeasured ampoules to each sample container

e. The same amount of preservative shall be added to the associated equipment blanks

- b. The pH shall be checked on all pH preserved samples (except VOC, O&G, and TRPH) using the following protocol:
 1. The effectiveness of required pH adjustment must be checked in the field.
 2. Narrow range pH paper shall be used to test an ALIQUOT of the preserved sample.
 - a. Pour a small portion of the sample into disposable container
 - b. Place the pH paper into the container and compare the color with the manufacturer's color chart
 - c. Discard the aliquot appropriately. DO NOT POUR BACK INTO THE SAMPLE CONTAINER.
 3. If the pH is acceptable (ex. greater than 10, less than 2, etc.), document acceptability in field records and prepare container for shipment to the laboratory.
 4. If the pH is unacceptable:
 - a. Add additional preservative in measured increments, mix and test ALIQUOTS of the sample as described above.
 - b. Continue to add measured increments of preservatives until an acceptable pH has been reached.
 - c. Record the TOTAL amount of preservative that was needed.
 - d. Additional chemical preservatives used in the field shall be from the same source as the chemical used for original preservation. DO NOT REUSE OLD SUPPLIES OF PRESERVATIVES.
 5. Alternatively, an extra "dummy sample" may be used used to test pH. Contents of the containers shall be suitably discarded.
 6. The same amount of additional chemical preservative shall be added to the corresponding equipment blank (or field blank, if used). NOTE: the maximum amount of preservative that was used to preserve any single sample in the sample set SHALL BE ADDED to the equipment blank.
 7. Sample pH shall be checked at the following minimum frequencies:
 - a. during the first sampling event at a particular site, ALL samples that are pH-adjusted must be checked, and
 - b. during subsequent visits to a particular site, AT LEAST ONE sample per parameter group that must be pH-adjusted shall will be checked.
 - c. If the frequency of sample collection at a specified location is greater than once per month (i.e. weekly or daily), the pH checks shall be made on AT LEAST ONE sample per parameter group according to the following schedule:

1. Weekly sampling - 1 pH check per month
 2. Daily sampling - 1 pH check per week
 3. < 1 sampling episodes per day - a minimum of 1 pH check, and 1 additional check per 10 sampling episodes.
- c. The organization preparing and preserving the containers must keep all documentation for preservation, consisting of:
- a. the grades and lot numbers of all preservation reagents
 - b. the opening date and expiration date.
 - c. the specific preservation technique that was used with each sample.

4.4.2.3 Holding Times, Container Types and Preservation

Holding time, container type and required preservation for samples shall comply with the following tables:

- a. Table 4.2 (Table II of 40 CFR Part 136) and Table 4.3 (Table 4 of Chapter 17-160, F.A.C.) for aqueous samples;
- b. Table 4.4 (Table 5 of Chapter 17-160, F.A.C.) for solid samples AND
- c. Table 4.5 (Table 8 of Chapter 17-160, F.A.C.) for specific drinking water holding times.

4.4.2.4 Special preservation protocols:

- a. All special preservation protocols outlined in Section 4.2.2 of the surface water section shall be followed for all aqueous samples.
- b. Samples for Chlorophyll shall be treated as follows:
 1. samples shall be filtered in the laboratory within 24 hr. of collection,
 2. magnesium carbonate shall be added to the filter while the last of the filtrate passes through the filter,
 3. the sample will be either analyzed immediately or frozen for later analysis within 21 days.

4.4.3 Sample Dispatch

4.4.3.1 Documentation

Field documentation will consist of, at a minimum, field notes, sample labels and Chain of Custody forms (or sample transmittal forms). These items must contain a minimum amount of information that can be traceable back to the original sampling event. A complete discussion of the mandatory information to be completed in the field are in Chapter 5, Sample Custody and Documentation.

TABLE 4.2
40 CFR Part 136 TABLE II: REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
(WATER/WASTEWATER SAMPLES)

PARAMETER NUMBER/NAME	CONTAINER(1)	PRESERVATION(2,3)	MAX HOLD TIME(4)
Table 1A-Bacterial Tests:			
1-4. Coliform, fecal and total	P, G	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5)	6 hours
5. Fecal streptococci	P, G	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5)	6 hours
Table 1B-Inorganic Tests:			
1. Acidity	P, G	Cool 4C	14 days
2. Alkalinity	P, G	Cool 4C	14 days
4. Ammonia	P, G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
9. Biochemical oxygen demand	P, G	Cool 4C	48 hours
11. Bromide	P, G	None required	28 days
14. Biochemical oxygen demand carbonaceous	P, G	Cool 4C	48 hours
15. Chemical oxygen demand	P, G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
16. Chloride	P, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze immediately
21. Color	P, G	Cool 4C	48 hours
23-24. Cyanide, total and amenable to chlorination	P, G	Cool 4C, NaOH to pH>12, 0.6g ascorbic acid (5)	14 days (6)
25. Fluoride	P	None required	28 days
27. Hardness	P, G	HNO ₃ to pH<2, H ₂ SO ₄ to pH<2	6 months
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately
31, 43. Kjeldahl and organic nitrogen	P, G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
Metals: (7)			
18. Chromium VI	P, G	Cool 4C	24 hours
35. Mercury	P, G	HNO ₃ to pH<2	28 days
3. 5-8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 45, 47, 51, 52, 58-60, 62, 63, 70-72, 74, 75. Metals, except chromium VI and mercury	P, G	HNO ₃ to pH<2	6 months
38. Nitrate	P, G	Cool 4C	48 hours
39. Nitrate-nitrite	P, G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
40. Nitrite	P, G	Cool 4C	48 hours
41. Oil and grease	G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
42. Organic carbon	P, G	Cool 4C, HCl or H ₂ SO ₄ to pH<2	28 days
44. Orthophosphate	P, G	Filter immediately, Cool 4C	48 hours
46. Oxygen, Dissolved Probe	G (Bottle & top)	None required	Analyze immediately
47. Oxygen, Winkler	G (Bottle & top)	Fix on site and store in dark	8 hours
48. Phenols	G only	Cool 4C, H ₂ SO ₄ to pH<2	28 days
49. Phosphorus (elemental)	G	Cool 4C	48 hours

TABLE 4.2, continued
40 CFR Part 136 TABLE II: REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
(WATER/WASTEWATER SAMPLES)

PARAMETER NUMBER/NAME	CONTAINER(1)	PRESERVATION(2,3)	MAX HOLD TIME(4)
50. Phosphorus, total	P, G	Cool 4C, H ₂ SO ₄ to pH<2	28 days
53. Residue, total	P, G	Cool 4C	7 days
54. Residue, Filterable	P, G	Cool 4C	7 days
55. Residue, Nonfilterable (TSS)	P, G	Cool 4C	7 days
56. Residue, Settleable	P, G	Cool 4C	48 hours
57. Residue, volatile	P, G	Cool 4C	7 days
61. Silica	P	Cool 4C	28 days
64. Specific conductance	P, G	Cool 4C	28 days
65. Sulfate	P, G	Cool 4C	28 days
66. Sulfide	P, G	Cool 4C add zinc acetate plus sodium hydroxide to pH>9	7 days
67. Sulfite	P, G	None required	Analyze immediately
68. Surfactants	P, G	Cool 4C	48 hours
69. Temperature	P, G	None required	Analyze immediately
73. Turbidity	P, G	Cool 4C	48 hours

Table 1C-Organic Tests:(8)

13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 56, 66, 88, 89, 92-95, 97. Purgeable Halocarbons	G, Teflon-lined septum	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5)	14 days
6, 57, 90 Purgeable aromatic hydrocarbons	"	Cool 4C, 0.008% Na ₂ S ₂ O ₃ , (5) HCl to pH2 (9)	14 days
3, 4, Acrolein and acrylonitrile	"	Cool 4C, 0.008% Na ₂ S ₂ O ₃ , (5) Adjust pH to 4-5 (10)	14 days
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96, Phenols (11)	G, Teflon lined cap	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction, 40 days after extraction
7, 38. Benzidines (11)	"	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5)	7 days until extraction, 40 days after extraction
14, 17, 48, 50-52. Phthalate esters (11)	"	Cool 4C	7 days until extraction, 40 days after extraction
72-74. Nitrosamines (11, 14)	"	Cool 4C, store in dark, 0.008% Na ₂ S ₂ O ₃ (5)	"
76-82. PCBs (11) acrylonitrile	"	Cool 4C	"
54, 55, 65, 69. Nitroaromatics and isophorone (11)	"	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5) store in dark	"
1, 2, 5, 8-12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons (11)	"	"	"
5, 16, 21, 31, 75. Haloethers (11)	"	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5)	"

TABLE 4.2, continued
40 CFR Part 136 TABLE II: REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
(WATER/WASTEWATER SAMPLES)

PARAMETER NUMBER/NAME	CONTAINER(1)	PRESERVATION(2,3)	MAX HOLD TIME(4)
29, 35-37, 60-63, 91. Chlorinated hydrocarbons (11)	"	Cool 4C	"
87. TCDD (11)	"	Cool 4C, 0.008% Na ₂ S ₂ O ₃ (5)	"
Table 1D-Pesticides Tests: 1-70. Pesticides (11)	"	Cool 4C, pH 5-9 (15)	"
Table 1E-Radiological Tests: 1-5. Alpha, beta and radium	P, G	HNO ₃ TO pH<2	6 months

TABLE II NOTES

1. Polyethylene (P) or Glass (G).
2. Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4 C until compositing and sample splitting is completed.
3. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO₃) in water solutions at concentrations 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under Part 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for shorter time if knowledge exists to show that this is necessary to maintain sample stability. See Part 136.3(e) for details.
5. Should only be used in the presence of residual chlorine.
6. Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
7. Samples should be filtered immediately on-site before adding preservative for dissolved metals.
8. Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
9. Sample receiving no pH adjustment must be analyzed within seven days of sampling.
10. The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

TABLE 4.2, continued
40 CFR Part 136 TABLE II: REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
(WATER/WASTEWATER SAMPLES)

11. When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4 C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).
12. If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0+/-0.2 to prevent rearrangement to benzidine.
13. Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
14. For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
15. The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na₂S₂O₃.

Reference: This table is reprinted from 40 CFR Chapter I, Revised as of July 1, 1988. According to Federal Register of Thursday, September 3, 1987, preservation for Oil and Grease may also be performed with ICL.

TABLE 4.3
 APPROVED WATER AND WASTEWATER PROCEDURES, CONTAINERS, PRESERVATION AND HOLDING TIMES
 FOR PARAMETERS NOT FOUND IN 40 CFR 136 **

PARAMETER	METHOD	REFERENCE (1)	CONTAINER (2)	PRESERVATION(3)	MAXIMUM HOLDING TIME(4)
Bromine	DPD Colorimetric(5)	SM 408E SM 4500-Cl-G	P, G	None required	Analyze immediately
Bromates	Ion Chromatography	EPA-300.0 B (6)	P, G	Cool, 4C	30 days
Chlorophylls	Spectrophotometric	SM 1002G SM 10200H	P, G (7)	14d in dark	30 days (7)
Corrosivity (CaCO ₃ Stability, Langelier Index)	Calculated	SM 203 SM 2330 ASTM D513-82	P, G	Cool, 4C (8)	7 days (8)
Odor	Human Panel	SM 207 SM 2150	G only	Cool, 4C	6 hours
Salinity	Electrometric (9) Hydrometric Argentometric	SM 210A SM 2420 B SM 2108 SM 2520 C SM 210C	G, wax seal	Analyze immediately or use wax seal	30 days (9)
Taste	Human Panel	SM 211 A,B SM 2160 B SM 2160 C SM 2160 D ASTM 1292-86	G only	Cool, 4C	24 hours
Total Dissolved Gases	Direct-Sensing Membrane-Diffusion Method	SM 2810	---	---	Analyze in-situ
Transparency	Irradiometric (10)	17-3.021(6) FAC	---	---	Analyze in-situ
Un-ionized Ammonia	Calculated (12)	DER-SOP (11)	P, G	Cool, 4C Na2S2O3 (12)	8 hours unpreserved 28 days preserved (12)
Organic Pesticides(13)	GC and HPLC	EPA (600-series) (14) (13)		(14)	(14)

**Reference: 17-160.700, F.A.C., Table 4

TABLE 4.3, continued
APPROVED WATER AND WASTEWATER PROCEDURES, CONTAINERS, PRESERVATION AND HOLDING TIMES
FOR PARAMETERS NOT FOUND IN 40 CFR 136

NOTES:

1) SM XXX = procedures from "Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 16th Edition, 1985 (except Chlorophylls, SM 1002G).

SM XXXX = procedures from Standard Methods for the Examination of Water and Wastewater", APHA-AWWA-WPCF, 17th Edition, 1989 (Except Chlorophylls, SM 10200H).

ASTM XXXX-YY= procedure from "Annual Book of ASTM Standards", Volumes 11.01 and 11.02 (Water I and II), 1988.

2) P= plastic, G= glass.

3) When specified, sample preservation should be performed immediately upon sample collection.

4) The times listed are the maximum times that samples may be held before analysis and still be considered valid.

5) The approved procedure is for residual chlorine. However, in the absence of chlorine, the DPD colorimetric procedure can be adapted to measure bromine content of the sample. In such case, the validity of this assumption must be verified by using another procedure for chlorine which is not affected by the presence of bromine (i.e. negligible interference).

(6) "The Determination of Inorganic Anions in Water Ion Chromatography", EPA Method 300.0 B, Revised August 1991, by John D. Pfaff, Carol A. Brockoff and James W. O'Dell, U.S. EPA, Cincinnati, Ohio 45268.

(7) Collect samples in opaque bottles and process under reduced light. Samples on filter taken from water having pH 7 or higher may be placed in airtight plastic bags and stored frozen for up to three weeks. Samples from acidic water must be processed promptly to prevent chlorophyll degradation.

(8) Temperature and pH must be measured on site at the time of sample collection. 7 days is the maximum time for laboratory analysis of total alkalinity, calcium ion and total solids.

(9) The electrometric and hydrometric analytical methods are suited for field use. The argentometric method is suited for laboratory use. Samples collected for laboratory analysis, when properly sealed with paraffin waxed stopper, may be held indefinitely. The maximum holding time of 30 days is recommended as a practical regulatory limit.

(10) Transparency in surface waters is defined as a compensation point for photosynthetic activity, i.e. the depth at which one percent of the light intensity entering at the water surface remains unabsorbed. The DER Rule 17-3 FAC requires that the light intensities at the surface and subsurface be measured simultaneously by irradiance meters such as the Kahlsico Underwater Irradiometer, Model No. 268 WA 310, or an equivalent device having a comparable spectral response.

(11) DER Central Analytical Laboratory, Tallahassee, FL, Revision No. 1, October 3, 1983. The 1983 draft is available from the DER QA Section.

(12) The results of the measurements of pH, temperature, salinity (if applicable) and the ammonium ion concentration in the sample are used to calculate the concentration of ammonia in the unionized state. Temperature, pH and salinity must be measured on site at the time of sample collection. Laboratory analysis of the ammonium ion concentration should be conducted within eight hours of sample collection. If prompt analysis of ammonia is impossible, preserve samples with H₂SO₄ to pH between 1.5 and 2. Acid-preserved samples, stored at 4C, may be held up to 28 days for ammonia determination. Sodium thiosulfate should only be used if fresh samples contain residual chlorine.

(13) Other pesticides listed in approved EPA methods (608.1, 608.2, 614, 614.1, 615, 617, 618, 619, 622, 622.1, 627, 629, 631, 632, 632.1, 633, 642, 643, 644 and 645) which are not included in Table ID of 40 CFR Part 136 (July 1989).

(14) Container, preservation and holding time as specified in each individual method shall be followed.

TABLE 4.4
 RECOMMENDED SAMPLE CONTAINERS, SAMPLE VOLUMES, PRESERVATION TECHNIQUES
 AND HOLDING TIMES FOR RESIDUALS, SOIL AND SEDIMENT SAMPLES (1)

PARAMETER GROUP	METHODS	REFERENCES	CONTAINER	PRESERVATION	MAX HOLDING TIMES
Volatile Organics	Purge-and-Trap GC and GC-MS	8010, 8015, 8020, 8021, 8230, 8240, 8260	Glass, 40 ml vial or 4 oz. widemouth with Teflon/silicone septum (2)	(3)	14 days
Semivolatile Organics	GC, HPLC, and GC-MS	8040, 8060, 8080, 8090 8100, 8120 8140, 8150, 8250, 8270, 8310	Glass, 8 oz. widemouth with Teflon lined cap (50 grams sample)	(3)	14 days until extraction, 40 days after extraction.
Total Metals-except mercury and chromium VI	Flame AA, Furnace AA, Hydride and ICP	All 7000-series methods (except 7195, 7196, 7197, 7198 7470, and 7471) and 6010 (ICP)	Glass or plastic, 8 oz. widemouth (200 grams sample)	(3)	6 months
Chromium VI	Colorimetric, Chelation with Flame AA	7196 and 7197	Glass or plastic, 8 oz. widemouth (200 grams sample)	(3)	24 hours
Mercury	Manual Cold Vapor AA	7471	Glass or plastic, 8 oz. widemouth (200 grams sample)	(3)	28 Days

(1) Adapted from tables 3-1 and 4-1 in Test Methods for Evaluating Solid Waste, SW-846, EPA, Third Edition, 1986, and First Update in 1987. The term residuals includes: (i) concentrated waste samples and (ii) sludges of domestic or industrial origin.

(2) Sample shall not be homogenized (mixed) prior to filling container. Container must be filled by packing as much sample into it leaving minimal headspace. Field samples can not be composited for analysis.

(3) Soils, sediments and sludges shall be kept cool at 4 C from collection time until analysis. No preservation is required for concentrated waste samples.

Reference: Chapter 17-160.700, F.A.C., Table 5

TABLE 4.5
PRESERVATION METHODS AND HOLDING TIMES FOR DRINKING WATER SAMPLES
THAT DIFFER FROM 40 CFR PART 136, TABLE II

Parameter	Drinking Water		40 CFR Part 136 Table II	
	Preservation(1)	Holding Time(2)	Preservation(1)	Holding Time(2)
Microbiologicals(3)	Cool 4 C Na2S2O3 (5)	30 Hours(4)	Cool 4 C Na2S2O3 (5)	6 hours
Radiologicals				
Group A(6)	HCl, HNO3 pH <2	6 months	HNO3 pH <2	6 months
Group B(7)	None	6 months	NA	NA
Nitrate				
Chlorinated(8)	Cool 4 C	28 days	Cool 4 C	48 hours
Non-Chlorinated(8)	H2SO4 pH <2	14 days(9)	Cool 4 C	48 hours

Footnotes:

1. Preservation, when required must be done upon sample collection.
2. Stated values are the maximum regulatory holding times.
3. Parameters included are: Fecal Coliform, Total Coliform and Fecal Streptococci.
4. If samples are analyzed after 30 hours, but within 48 hours of collection, the laboratory is to indicate in the analytical report that the data may be invalid because of excessive delay in sample processing. No samples received after 48 hours are to be accepted or analyzed for compliance with the regulations of the Department of Environmental Regulation or the Department of Health and Rehabilitative Services.
5. Addition of sodium thiosulfate is only required if the sample has a detectable amount of residual chlorine, as indicated by a field test using EPA Method 330.4 or 330.2 or equivalent.
6. Group A parameters are: Gross Alpha, Gross Beta, Strontium-90, Radium-226, Radium-228, Uranium and Photon Emitters.
7. Group B parameters are: Cesium-134, Iodine-131, and Tritium.
8. Chlorinated means that the source water has a detectable amount of residual chlorine, as indicated by a field test using EPA Method 330.4 or 330.2 or equivalent. Non-chlorinated means that the source water contains no detectable amount of residual chlorine (i.e. is below the method detection limit of the above-mentioned methods).
9. Ion chromatographic methods using conductivity as the detector cannot be used to analyze for nitrate in such samples.

Reference: Chapter 17-160.700, F.A.C., Table 8

4.4.3.2 Sample Packing and Transport

a. Samples shall be packed such that they are segregated by site, sampling location or by sample analysis type. Sample segregation may follow this segregation scheme or any other that is sensible and well thought out. These schemes are dependent upon the levels of contamination present, the number of bottles to be transported, the size of the bottles, etc.

1. VOC samples from different locations may be placed into the same cooler to reduce the number of required trip blanks provided that the samples are wrapped or containerized (ziplock bag or metal can) separately.

2. Samples in breakable containers shall be packed with materials (i.e. bubble wrap, cans with vermiculite) to avoid breakage.

3. Shipping transport containers shall be insulated (if cooling is required).

4. Shipping containers shall be sealed with strapping tape or locked to avoid tampering. Tamper-proof seals may also be placed over cooler lid.

5. All samples that require thermal preservation shall be packed in thermally insulated coolers with wet ice. Only wet ice shall be used in cooling samples to 4 C. BLUE ICE OR CHEMICAL COOLING PACKS ARE NOT ACCEPTABLE.

b. Packed samples shall be delivered to the analyzing laboratory by the sampling team or via common carrier.

1. If sent by common carrier, all documentation (transmittal form, bill of lading, analyses order, etc.) shall be sealed and placed inside the shipping container prior to sealing it closed. The QA Section recommends that all records be placed in a plastic bag and taped to the underside of the cooler lid.

4.4.4 Field Reagent Handling

All reagents, cleaning materials and preservatives that are maintained by the field consultant shall be stored, transported and handled in such a way to prevent and/or minimize contamination.

The following storage and use protocols shall be observed. Those that are double bracketed ([[]]) are RECOMMENDED protocols that should be considered when storing various chemicals:

[[a. All chemicals that are maintained in-house and transported to the field shall be segregated according to reactivity (i.e. acids, bases, etc.).]]

[[b. If possible, acids should be stored in an acid storage cabinet and solvents should be stored in a vented solvent storage cabinet. If specialized storage is unavailable, all chemicals shall be stored in a well-ventilated area.]]

c. All chemicals transported to the field shall be stored in bottles which will be packed to avoid breakage.

d. If quantities of reagent chemicals are transferred from the original container, the transport container shall be appropriately precleaned and must be of similar construction type as the original container (e.g. acids and bases may be transported in plastic or teflon containers).

e. Chemicals shall be segregated from sample containers so as to avoid reaction and accidental contamination.

[[f. Acids and bases must be segregated to prevent reaction.]]

g. Analyte-free water shall be segregated from solvents to prevent contamination.

4.4.5 Field Waste Disposal

4.4.5.1 General Considerations

Field-generated wastes may require segregation and containerization for proper disposal by a commercial contractor. This decision is highly dependent upon which DER program the site work is being conducted and the nature of the waste. If there is a DER technical Project Manager that is in an oversight position, they should be consulted regarding the fate of all wastes. In general, these wastes can be categorized as: (1) decontamination wastes, especially waste solvents, (2) waste acids and bases, (3) contaminated purge waters, and (4) calibration standards from field meters.

a. All field investigations will generate some amount of waste material, especially groundwater investigations. Boring, developing, purging, and sampling monitor wells will generate soils, waters, and spent reagents that must be handled in a way that will not spread or increase contamination at the site. Activities from other sampling matrices will generate similar wastes.

b. These wastes are normally categorized into hazardous and non-hazardous wastes.

1. Hazardous wastes must be disposed of according to any and all applicable Federal (RCRA, CERCLA, etc.), State, County, or municipal regulations.

2. Non-hazardous wastes must also be disposed of appropriately.

c. Proper handling and disposal of all waste materials should be addressed prior to initiating site work. The following are a list of some materials that will require proper treatment, storage, and disposal. Additionally, these must be segregated by their hazardous or non-hazardous nature:

1. personnel equipment: coveralls, gloves, boots, suits, disposable booties,
2. disposable equipment: ground covers, equipment covers (aluminum foil, plastic garbage bags, etc.), disposable bailers or tubing, broken or unused sample containers, shipping containers, etc.,
3. soil cuttings from drilling or hand boring,
4. drilling mud or fluid,
5. development and purge waters,
6. decontamination wastes: spent solvents and acids, and
7. spent calibration standards for field analytical equipment (field GC, conductance, pH, etc.).

4.4.5.2 Decontamination Wastes

a. Decontamination and calibration wastes must be segregated and disposed of properly.

b. Soap solutions and waste tap/DI/analyte-free water can be disposed of on site.

c. Calibration standards (pH and conductance) may be diluted with spent detergent solution and wash waters and disposed of in sanitary sewer.

d. Weak acid solutions may be neutralized or diluted and disposed of properly.

e. Waste solvents shall be handled as hazardous waste and must be collected and transported back to the office or lab to be handled by commercial disposal or recycling contractor. DISCHARGE OR EVAPORATION OF WASTE SOLVENTS ON-SITE, IN ANY AMOUNT, IS NOT ACCEPTABLE. See discussion below on hazardous waste handling.

f. Concentrated, reagent grade preservative acids and bases shall be transported back to the office, laboratory, or disposed of by commercial contractor.

g. Field GC standards must be handled as a waste solvents.

4.4.5.3 Disposal of purged water

- a. Contaminated purge waters must be handled prudently. Recommendations from an DER Project Manager should be used and will supersede guidance defined in this document.
- b. If wastewater generated from well development or purging of monitor wells is likely to contain contaminants in excess of the MCL (maximum contaminant level) as listed in Florida Administrative Code (e.g. Chapter 17-302, 17-550 or 17-770), the water shall be contained on-site in temporary storage (e.g., lined pit, drum, tank or tanker truck) until the waters can be characterized by the appropriate approved analytical method(s).
- c. In many cases it may be possible to directly discharge contaminated purged water on-site, but only if the purged water will infiltrate into the SAME aquifer zone from which it was purged from or into a more contaminated aquifer zone.
- d. Additionally, exposure of such purged water must not pose a health risk and the purged water shall not be discharged into any surface water body, unless permitted.
- e. Purged water must be adequately treated (contaminants should not exceed established standards) prior to discharge on-site if the above conditions cannot be met.
- f. Alternatively, purged water may be transported to an off-site facility such as sewage treatment plant/sewer system (some wastewater treatment systems are capable of treating water with total VOC concentrations up to 500 ppm). THE WASTEWATER TREATMENT PLANT OPERATOR MUST BE NOTIFIED AND MUST GIVE APPROVAL BEFORE DISCHARGE CAN OCCUR.
- g. The QA Section strongly recommends contacting the overseeing regulatory authority concerning criteria for discharge of moderately contaminated purge water. By contacting the DER (District or headquarters), county, or municipal authority that is in charge of a particular program, local ordinances or procedures may be obtained that relate to collection and transportation of purge water.

4.4.5.4 Field Generated Hazardous Waste

Handling, storage and disposal of field-related hazardous wastes are subject to the regulations contained in the Resource Conservation and Recovery Act.

All of the procedures listed in this subsection pertain to the company, field consultant, primary contractor, etc. whomever is performing and is responsible for the field sampling event. For brevity, we will refer to this organization as the field consultant.

a. Responsibilities

1. The field consultant is responsible for all wastes generated on-site as a result of the sampling event, excluding those waste materials already present on-site (contaminated drill cuttings, purge water, etc.).

2. It is the responsibility of the field consultant to store, package, label, ship and dispose of the hazardous wastes which are generated during the sampling event or project in a manner which ensures compliance with all Federal, State and local laws, regulations and ordinances.

3. Responsibility may also be assumed by the property owner. These requirements will not specify who is ultimately responsible. This decision will be made by the property owner and the primary contractor with regard to ALL of the RCRA requirements.

4. The field consultant is responsible for the waste if it contaminates the environment; therefore, precautions should be taken to secure all reagents (acids, bases, solvents, etc.) that, if spilled, would be characterized as a hazardous waste (listed in 40 CFR Part 261.30-.33 or if a characteristic waste).

b. Definitions

1. A hazardous waste can be defined by any one of the following criteria;

a. The waste material is listed in 40 CFR Part 261.30-261.33.

b. The material exhibits any of the specified characteristics: ignitability; corrosivity; reactivity or TC toxicity.

2. Classification

a. Field consultants that generate hazardous waste are put into 3 categories based on the amount of hazardous waste generated monthly. These categories are; 1) conditionally exempt small quantity generator, 2) small quantity generator and 3) full generator.

b. Conditionally Exempt Small Quantity Generator:
A generator who generates no more than 100 kilograms of hazardous waste in a calendar month. (40 CFR Part 261.5)

c. Small Quantity Generator:

A generator who generates more than 100 kilograms but no more than 1000 kilograms of hazardous waste per calendar month or generates less than 1 kilogram of acute hazardous waste and accumulates no greater than 6000 kilograms of hazardous waste. (40 CFR Part 262.34)

d. Full Generator:

A generator who generates wastes in excess of 1000 kilograms per calendar month or more than 1 kilogram per month of acute hazardous waste. (40 CFR Part 262.34).

3. It is the responsibility of the field consultant to know which category their organization falls under. Since most field consultants will fall into the conditionally exempt small quantity generator category, these requirements are listed below.

c. Hazardous waste Handling Protocols for Conditionally Exempt Small Quantity Generators

1. These generators may either treat or dispose of hazardous waste in an on-site facility or ensure delivery to an off-site treatment, storage or disposal facility, either of which, if located in the U.S., is:

- a. Permitted under Part 270 of the federal regulations
- b. In interim status under Parts 270 & 265.
- c. Authorized to manage hazardous waste by a state with a hazardous waste management program approved under Part 271.
- d. Permitted, licensed, or registered by a state to manage municipal or industrial solid waste.
*(subject to local regulations).

d. Hazardous Waste Handling Protocols for Facilities falling into the Small Quantity Generator and Full Generator

1. These organizations must adhere to all regulations pertaining to waste disposal in the Resource Conservation and Recovery Act.

e. General Disposal/Treatment Considerations:

1. Hazardous waste solvents, as identified in the 40 CFR Part 261, shall not be evaporated on-site by pouring onto pervious or impervious surfaces.

- a. These solvents shall also not be evaporated at the office or lab with or without a fume hood.
- b. Solvents that evaporate during the actual decontamination process are exempt.

2. Acidic and Basic wastes may be neutralized and disposed of via the sanitary sewer if they are not hazardous due to the presence of other constituents*. (*subject to local regulations).

f. Transportation

1. There are no special handling requirements for transportation of these wastes back to the office or laboratory.

2. There are no requirements for manifesting the waste nor placarding the vehicle (if for small quantities).

3. A sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements.

g. Storage and Accumulation:

1. Hazardous waste storage is limited by quantity and/or accumulation time and must comply with RCRA regulations as specified in the 40 CFR.

2. These wastes shall be packaged and separated according to compatible groups (e.g. solvents, acids, etc.).

h. Sample Disposal:

1. Samples submitted to a laboratory for analysis are excluded from regulation as hazardous waste under 40 CFR Part 261.4(d) provided the samples are being transported to or from the laboratory, or are being analyzed, are being held for analysis, or are being maintained in custody for legal reasons.

2. Once a decision is made to dispose of laboratory samples, the exclusion provisions of 40 CFR Part 261.4(d) no longer apply.

3. Samples that have been identified as hazardous may either be: 1) returned safely to the generator; or 2) disposed of according to applicable RCRA regulations summarized in this document.

4. Samples which are determined to be non-hazardous may be subject to local environmental regulations. It will be the responsibility of the laboratory to be familiar with any such local regulations.

5.0 SAMPLE CUSTODY AND DOCUMENTATION

The following discussions outline the minimum record keeping requirements as they relate to sample collection, sample handling and sample analysis activities. The protocols and requirements outlined in this section emphasize the use of unequivocal, accurate and methodical records to document all activities affecting sample data.

Additional requirements may be imposed by specific DER programs (e.g. Drinking Water) or other agencies (e.g. HRS, EPA, etc.). If applicable, the organization shall meet the DER QA requirements and the additional or more stringent requirements of the other programs or agencies.

There are two levels of custody: 1) Sample custody or tracking and 2) Legal or evidentiary chain of custody.

1. Sample custody or tracking is required by DER. It includes all records and documentation necessary to trace a sample from point of origin through final report and sample disposal. Sample custody requires that each event or procedure to which the sample is subjected be documented. These include, but are not limited to: sample collection, field preservation, sample receipt and log in, sample preparation, sample analysis and sample disposal. In addition, those tasks or activities that relate to each of the above-mentioned events (e.g. reagent preparation, calibration, preventative maintenance, quality control measures, etc.) must be documented. The history of the sample must be readily understood through the documentation. The required documentation that is associated with sample custody is outlined in Sections 5.1 through 5.5.

2. Legal or Evidentiary Chain of Custody (COC) is a special type of sample custody which requires that the physical possession, transport and storage of a sample be documented in writing. The records must account for all periods of time from sample container acquisition through sample disposal. COC protocols are not required by the Department, but are recommended. If implemented, the minimum documentation requirements outlined in Section 5.6 must be followed.

5.1 GENERAL REQUIREMENTS FOR CUSTODY AND DOCUMENTATION

5.1.1 Record Keeping System Design - General Requirements

Each organization shall design and maintain a record keeping system that is succinct and efficient:

1. All records shall be maintained in a manner which facilitates documentation tracking and allows historical reconstruction of all analytical events and ancillary procedures that produced the resultant sample analytical data.

2. The system shall unequivocally link all documentation associated with a sampling event from sample collection through the final analytical result and sample disposal. This may be accomplished through either direct or cross-references to specific documentation.

3. The system shall be straightforward and shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

4. Final reports, data summaries, or other condensed versions of data that have been prepared by external parties shall be linked to internal records by an unequivocal cross-referencing mechanism (usually field and/or laboratory ID numbers).

5.1.2 Documentation Criteria

1. The history of a sample must be clearly evident from the retained records and documentation. Copies or originals of all documentation which are associated with the analysis or sample collection event must be kept. This includes the documentation that is sent to or received from all sampling and analysis organizations.

2. All applicable documentation specified in this section shall be available for inspection during any sampling-site, facility (laboratory or offices) or data audit conducted by authorized representatives of DER.

3. The records must contain enough information so that excessive clarifications, interpretations or explanations of the data are not required from the originator.

4. All documentation and record entries shall clearly indicate the nature and intent of each entry.

a. All documentation entries shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records (e.g. sampled by; prepared by; reviewed by, etc.).

b. Often, documentation requirements can be met by making brief references to procedures written in internal SOPs or approved methodology promulgated by external sources. If these standard procedures are routinely repeated in your

operations (e.g., sample preparation procedures, decontamination protocols, analytical method, etc.), then citing these references may be appropriate. Such citations must specifically identify the document, method or SOP (e.g. sample preparation by 3010; field decon per internal SOP for teflon sampling equipment, etc.), and must include the revision number or revision date. Copies of all revisions must be retained as part of the laboratory documentation.

5.1.3 Record-keeping Protocols

1. Entries into all records shall be made with waterproof ink.
2. Entries in records shall not be obliterated by erasures or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction.

5.2 PREPARATION OF FIELD SAMPLING SUPPLIES AND EQUIPMENT

All parties providing sample containers, preservation reagents or sampling equipment shall maintain tracking records.

A system of records or codes shall be designed to link cleaning records, preservation or reagent preparation records and trip blanks (if applicable) to the associated equipment, containers, prepreserved containers, analyte-free water and preservatives which may be shipped in sampling kits.

These records shall be maintained by the party responsible for providing any or all of the above-mentioned equipment, containers and/or reagents.

5.2.1 Content Requirements For Sampling Kit Documentation

The contents of each prepared sampling kit (see Appendix A for definition) shall be documented. A packing list or similar record shall be transmitted to the receiving party with the sampling kit and a copy or other record shall be retained by the preparing party.

5.2.1.1 The following information shall be transmitted to the receiving party:

- a. Quantity, description and material composition of all containers, container closures or closure liners (if method specified) and all sampling equipment;
- b. Intended application for each container type indicated by approved analytical method or method group;
- c. Type and concentration of preservative added to clean sample containers and/or shipped as additional preservative;

- d. Intended use of any additional preservatives or reagents;
- e. Description of any analyte-free water (i.e. deionized, organic-free, etc.);
- f. Types and number of any quality control blanks (e.g., trip blanks);
- g. Date of kit preparation; and
- h. Description and material composition of all reagent transfer implements, e.g., pipets, shipped in the kit.

This information may be in the form of a packing slip (e.g., 6-125 ml plastic containers for metals, 12 VOC vials for 601/602, etc.).

5.2.1.2 In addition to maintaining records of the above information, the preparing party shall maintain records or cross reference links of the following information:

- a. Lot numbers of any commercially obtained sources of analyte-free water (if provided);
- b. Material composition of all reagent and analyte-free water containers (if provided);
- c. A code or reference (i.e., lot numbers) to dates in container and/or equipment cleaning logs;
- d. A code or reference that links preservatives to preparation logs for preservatives or vendor lots;
- e. Name of receiver of kit;
- f. Project name for kit use, if known;
- g. Name of individual(s) preparing the kit; and
- h. Date the kit was shipped or provided.

5.2.1.3 If the sampling kits are prepared for internal use (i.e. they will not be shipped to any external party, including branch offices of the same organization) and the sampling kits are used for collecting routine (i.e. daily, weekly or monthly monitoring) samples, the records in 5.2.1.1 and 5.2.1.2 may be reduced to the following:

- a. The cleaning records for sampling equipment and/or sample containers (see 5.2.3 below) shall indicate who received the cleaned containers or equipment and the date of receipt.
- b. The preservation and/or reagent preparation records shall indicate that the preservative or reagent was prepared for use in the field.

5.2.2 Documentation for Preservatives

Sample preservatives and other reagent preparations shall be traceable to preparation dates and vendor sources and/or lot numbers.

5.2.3 Documentation of Cleaning Procedures for Sampling Equipment and Containers for Samples, Reagents and Analyte-Free Water

Records shall be maintained for all container or equipment cleaning. This requirement shall apply to all containers and equipment cleaned or prepared for use in field sampling activities. Field-meter probes or other devices contacting the sample or sample source are also included in this category:

1. The material composition, size and any other description of all sample, preservative or analyte-free water containers, container closures and closure liners or sampling equipment cleaned or prepared;
2. A detailed, step-by-step description of the cleaning protocols including cleaning agents, water types or other reagents used in the procedure. Reference to internal SOPs may be used (see 5.1.2.4.b);
3. Date of cleaning;
4. Individual(s) responsible for cleaning;
5. Storage conditions (i.e. wrapped in foil, boxed, etc.) shall be described (internal SOPs may be referenced);
6. Storage location for cleaned containers and equipment;
7. Unique ID numbers or codes assigned to individual containers or pieces of equipment;
8. The number of cleaned containers or equipment if prepared or cleaned in groups or lots (i.e., cleaned or prepared in the same session, using the same lots of cleaning materials). A group or lot number may be assigned. This code must be linked to any individual IDs (if used), the cleaning date (see 5.2.3.3 above) and any sampling kit IDs and records.
9. Any quality control information concerning the cleanliness of the containers and/or equipment must be retained and must be linked to the set (see 5.2.3.8).
10. The intended end use of the equipment and/or containers shall be indicated (e.g. Teflon bailers cleaned for organics; 30 plastic containers cleaned for metals, etc.).

5.3 CUSTODY AND DOCUMENTATION REQUIREMENTS FOR FIELD OPERATIONS

The following documentation requirements shall be followed for all field-sampling operations.

5.3.1 General Protocols

1. Copies of all COC forms (if applicable) or sample transmittal forms shall be maintained with project records. If the sampling and analysis activities are performed by the same organization at the same physical location (e.g. wastewater

sampling and analysis) and if all records are maintained in a central location, a single copy of the COC form (if used) or the laboratory transmittal form may be retained.

2. Entries into all field records shall be made with waterproof ink.

3. Errors in all documents shall be deleted with one line (see Section 5.1.3.2).

4. All documentation/logs shall be signed/initialed by the appropriate personnel.

5. It is recommended that all time be recorded using 24 hour notation (e.g., 2:00 PM is 1400 hours).

5.3.2 Sample Identification Requirements

1. All sample containers must be labeled (tagged).
 - a. At a minimum, the label or tag shall identify the sample with the field ID number.
 - b. Additional information (i.e. preservation, sampler's name, etc.) may be included as a part of the tag or label.
 - c. The label or tag shall be attached so that it does not contact any portion of the sample that is removed or poured from the container.

2. The Field ID number shall be a unique number or code that is assigned to EACH sample container. The assigned code must unequivocally link the collected sample to the time or date of sampling, and may include information concerning the location of the sampling point. Samples that are routinely collected from the same location (e.g. monitoring wells, outfalls, etc.) must be identified by more than the designated location code. For example:

- a. MW-1V does not adequately identify a VOC sample collected at monitor well 1;
- b. MW-1-392V would identify the sample as being collected from monitor well 1 in March 1992 for VOCs.

3. At a minimum, the ID numbers must be recorded on all sample tags (or labels), in the field records, and on all transmittal records or COC forms.

4. Ancillary records (photographs, videotapes, maps, etc.) must be easily traced to specific sampling events and are subject to the same custody requirements as other records discussed in this Section.

5.3.3 Required Documentation

All activities related to sampling events shall be documented in the field records. At a minimum, the types of records that must be maintained include, but are not limited to the following:

1. Sample labels/tags (with identifying ID#s).
2. Sample seals (if required)
3. Sample transmittal forms (or COC forms).
4. Field sheets, logs, notebooks or other records

5.3.4 Required Information

5.3.4.1 Sample Transmittal Records

All samples that are submitted to a laboratory must be accompanied by a sample transmittal or Chain of Custody record (see Section 5.6). This record may be designed as individual forms for each sample or a summary form for a set of samples. AT A MINIMUM, the information transmitted to the laboratory shall include:

- a. Site name and address (Client Code may be acceptable if samples are considered sensitive information and if the field records clearly trace the code to a specified site and address)
- b. Date and time (military time preferred) of sample collection
- c. Name of sampler responsible for sample transmittal
- d. Field ID#(s) (see 5.3.2 above)
- e. Number of samples
- f. Intended analyses - The analytical method number shall be listed if the sample results are related to a QAPP or other document (e.g. DER Rule or permit) which specifies the method to be used.
- g. Preservation (may be indicated on sample label/field sheets)
- h. Comments section (about sample or sample conditions)
- i. Appropriate place for identification of common carrier (if used)

5.3.4.2 Field Records

The following information must be documented in the records maintained by the sampling organization. This information may be recorded in bound notebooks or on field sheets that have been designed for a specific purpose. All loose records (i.e. field sheets, photographs, etc.) shall be unequivocally linked to the sampling event by code, facility name and/or client name and address.

- a. General Information - the following information shall be recorded for all sampling events:
 1. Names of all personnel and visitors on site during sampling
 2. Date and time (military time preferred) of sample collection
 3. Ambient field conditions, to include, but not limited to information such as weather, tides, etc.
 4. Specific description of sample location including site name and address. The specific sampling point must be further identified (Well ID #, outfall number, lat/longs, station number, etc.).
 5. Field ID# (see 5.3.2 above) for each sample container and parameters to be analyzed
 6. Field measurement data (e.g., pH, specific conductance, etc.)
 - a. Records shall indicate when measurements were taken; and
 - b. Calibration information to include: time of all calibrations or calibration checks, concentration(s) of standards and calibration acceptance (information may be kept in a separate calibration log)
 7. Sample sequence - identify the order in which each sample is taken (time of sample collection is acceptable). NOTE: if the collection time is used, the time that each sample aliquot is collected (i.e. VOC, metals, nutrients, etc.) MUST BE NOTED.
 8. Preservative used - information must include, but is not limited to:
 - a. Preservative name;
 - b. pH verification (if applicable)
 - c. Amount/quantity of preservative that is added (if adding preservatives in the field); and
 - d. Amount/quantity of additional preservative that is added (if using sample containers with premeasured preservatives)
 9. Purging and sampling equipment used (ID# if applicable)
 10. Field decontamination performed. All field-sampling equipment decontamination, whether performed in the field, on site or in a headquarters facility or laboratory, must be documented per 5.2.3 above.
 11. Types of QC samples collected. Include when and where collected, preservative (if applicable) and type (e.g., trip blank, equipment blank, duplicate, etc.). QC samples must be documented in the same manner as all other samples.

12. Use and location of fuel powered units (if applicable)

13. Composite samples (if collected) shall indicate number of samples in the composite and approximate amount/quantity of each subsample

14. Signature of sampler(s).

b. Additional documentation for monitoring wells shall include:

1. Well casing composition and diameter of well casing
2. Water table depth and well depth
3. Calculations used to determine purge volume
4. Total amount of water purged
5. Date well was purged
6. Beginning and ending purge times (military preferred)

7. Measurement data to monitor stabilization, if applicable (see 5.3.4.2.a.6 above)

8. Drilling/boring method (if known), including type/name of drilling mud used (if known) - These may be by reference to associated well logs or well installation records.

c. Additional documentation for in-place plumbing and/or drinking water sources shall include:

1. Plumbing and tap material construction (if known)
2. Flow rate at which well was purged
3. Time well was allowed to purge
4. Flow rate when sample collected
5. Public water system ID # (if applicable)
6. Name and address of water supply system as well as an emergency phone number for notification of sample results (if applicable)

d. Additional documentation for surface water shall include:

Depth samples were taken.

e. Additional documentation for wastewater effluent shall include:

1. Beginning and ending times (24 hr) for timed composite sampling
2. Type of composite (e.g. flow proportioned, continuous, etc.)

f. Additional documentation for sediments and soils shall include:

1. Depth from the surface that samples were taken

2. Drilling/boring method (if known), including type/name of drilling mud used (if known). See 5.3.4.2.b.8 above.

g. Additional documentation for drum sampling shall include:

1. Type of drum, description of contents and markings
2. If stratified, what layer(s) were sampled

5.3.5 Sample transport:

1. All sample transmittal forms shall be placed in waterproof bags and sealed in the transport containers with the samples.
2. If shipped by common carrier, transport containers should be securely sealed with strapping tape or other means to prevent lids from accidentally opening. COC Seals (if used) shall be applied after containers have been secured.
3. All shipping bills from common carriers shall be kept with the COC or transmittal forms.

5.4 SAMPLE CUSTODY TRACKING AND DATA DOCUMENTATION FOR LABORATORY OPERATIONS

5.4.1 Initial Check of Samples and Documentation

When samples are received by the laboratory the following checks shall be made upon receipt:

1. Verify the integrity and condition of all sample containers.
 - a. Check for leakage, cracked or broken closures or containers, evidence of grossly contaminated container exteriors or shipping cooler interiors, and obvious odors, etc.
 - b. Check for air headspace or bubbles in VOC containers.
2. Verify receipt of complete documentation for each container.
 - a. The minimum information FOR EACH SAMPLE CONTAINER must include the items listed in 5.3.4.1.
 - b. Verify that sample identification numbers on sample transmittal forms correspond to sample identification numbers on the sample containers.

5.4.2 Verification of Sample Preservation

1. Verify proper field preservation of each sample by examination of documentation received from the field-sampling party. There must be clear documentation of any chemical

preservation of the sample. This documentation must demonstrate proper preservation per approved preservation protocols listed in 4.4.2.

2. For samples which require thermal preservation (i.e. wet ice), verify proper storage temperature by determining that sample containers are in adequate contact with wet ice in the shipping chest. The following alternate techniques may be used to verify the actual sample temperature:

- a. The temperature may be verified by determining the temperature of a surrogate water sample which has been shipped with the samples or placed in the transport containers with the samples after arrival in the laboratory. In the latter case, the surrogate sample must be allowed to equilibrate to the temperature of the samples in the cooler.
- b. The temperature of incoming samples may also be verified by a non-invasive temperature probe.
- c. The temperature of the melted ice water in the cooler may also be used as an indicator of proper temperature.

UNDER NO CONDITIONS SHALL A THERMOMETER OR OTHER TEMPERATURE MEASURING DEVICE BE PLACED INTO THE COLLECTED SAMPLE CONTAINER.

3. For acid- or base-preserved samples, verify the pH of the samples at time of receipt in the laboratory or upon preparation of subsamples and other aliquots for analysis (see 5.4.8.3 below).

5.4.3 Rejection of Received Samples

1. Rejection Criteria - Samples shall be rejected according to the following criteria.

- a. The integrity of sample containers is compromised as described in 5.4.1
- b. The identification of a container cannot be verified
- c. The proper preservation of the container cannot be established
- d. VOC vials contain bubbles of sizes greater than 1% of the vial volume (usually a bubble size of 5 mm in diameter). Note: the presence of any bubbles in VOC vials must be documented and reported with the final results.

2. The laboratory shall obtain concurrence or further instruction from the sample submitter regarding any proposed rejection. All correspondence and/or conversations concerning the final disposition of the samples shall be documented.

3. Any decision to proceed with the analysis of compromised samples shall be fully documented.

- a. The condition of these samples shall be noted in all documentation associated with the sample.
- b. The analysis data shall be appropriately qualified as estimated on all internal documentation and on the final report (see Data Qualifiers, Table 10.2).

4. Rejected samples shall be logged in the laboratory sequential log per Section 5.4.4 below with appropriate comments.

5.4.4 Sample Receipt Logging

1. The laboratory shall employ a logical system for assigning a unique identification code to EACH SAMPLE CONTAINER received in the laboratory. Multiple aliquots of a sample that have been received for different analytical tests (e.g., nutrients, metals, VOCs, etc.) shall be assigned a different ID code.

- a. This laboratory code shall maintain an unequivocal link with the unique field ID assigned each container.
- b. The identification of containers by container shape or size is not adequate.
- c. Sample containers will be labeled with the unique code upon assignment of the code.

2. A chronological log shall be employed to document receipt of all sample containers. The following information will be recorded in the laboratory sequential log:

- a. date of laboratory receipt of sample
- b. sample collection date
- c. unique laboratory ID code
- d. field ID code supplied by sample submitter
- e. required analyses, including approved method number
- f. signature or initials of logger
- g. comments resulting from sample integrity inspection (Section 5.4.1) or sample rejection (Section 5.4.3).
- h. sampling kit code (if applicable)

3. Smaller laboratories whose function is to analyze on-site samples that have been collected by the laboratory staff (e.g. in-house domestic wastewater treatment laboratories) may use the sample transmittal forms as the sample log provided:

- a. The information in 5.4.4.2 above is included on the forms; and
- b. The sheets are maintained in chronological order as a permanent laboratory record.

In these cases, the laboratory ID number may be the same as the field ID number, subject to the requirements listed in Sections 5.4.4.1 and 5.4.4.2 above.

4. Retain all documentation that is transmitted to the laboratory by the sample transmitter.

5.4.5 Sample Storage

1. Parent samples, sample replicates and subsamples received in the laboratory shall be stored under approved conditions as described in Tables 4.2, 4.3, 4.4 and 4.5.

2. Sample fractions, extracts, eluates, leachates, digestates, etc. shall be stored according to requirements of 5.4.5.1 above or according to guidance found in the approved preparation or analytical method used to prepare or analyze the subsample, as applicable. In cases of conflicting guidance, the storage/preservation requirements specified in 5.4.5.1 above shall supersede method guidance. No specific requirements apply to other cases not comprised by the above.

3. Samples and all subsamples, sample fractions, extracts, eluates, leachates and digestates shall be stored separately from all standards, reagents, cleaning supplies, fuels, food, etc.

4. VOC samples shall be stored separately from all other samples.

5. The manner in which samples and subsamples are stored shall be documented. This may be recorded in the sample receipt log (5.4.4) or other linked documentation.

5.4.6 Sample Disposal

At a minimum, record the date of sample and/or subsample disposal and either the name (or initials) of the individual authorizing the disposal or the person who is responsible for the disposal.

5.4.7 Intralaboratory Distribution of Samples for Analysis

1. The laboratory shall utilize a proactive procedure to ensure that all samples and subsamples are analyzed within allowed maximum holding times (specified in 5.4.5.1 above).

2. All distribution of samples and subsamples for preparation and analysis shall be documented as to task assignment and analysis date deadline.

5.4.8 Laboratory Preparation of Samples for Analysis

Record all sample preparation procedures that may impact the analytical results.

5.4.8.1 Preparation Records

- a. Sample preparation records shall include, but are not limited to:
 1. digestions
 2. filtrations
 3. distillations
 4. extractions
 5. leachings
 6. sample extract cleanup procedures
- b. The specific sample processing protocol shall be identified. Where the procedure is routinely performed according to approved methodology or internal SOPs, preparation records may refer to the specific method or SOP (see 5.1.2.4.b)

5.4.8.2 Required information

- a. All numerical parameters associated with the preparation technique shall be recorded. These data shall include, but are not limited to:
 1. Sample or subsample ID number
 2. Duration times for processes (e.g., extraction cycles, digestions, distillations, sonications, etc.) if the method specifies a time limitation
 3. Volumes or weights of subsamples, reagents or dilution water
 4. Dilution factors
 5. Meter and other instrument readings
 6. Chromatography column elution profile retention times
 7. Adsorption column efficiency or breakthrough determinations
 8. pH checks
- b. Where specific materials or supplies are explicitly required by the approved method, record description and the material composition of such equipment, labware or supplies. This information may be by reference to internal standard operating procedures (see 5.1.2.4.b).
- c. Record all calculations associated with the preparation procedure.
- d. Retain all elution profile chromatograms, pH meter recorder charts or other products of automatic instrument data recordings associated with the procedure.
- e. Link all reagents that are used in the procedure to the applicable reagent preparation records.

5.4.8.3 pH Checks of Samples and Subsamples

- a. Verify the pH of all pH-preserved samples before any sample preparation or sample analysis procedure.

Additional pH checks and adjustments, where required by the approved method, shall be documented.

b. Record the results of pH checks on samples and subsamples.

c. The proper pH value as stipulated by approved preservation protocols or approved sample preparation methods shall follow the method prescribed procedures. If none are specified, the pH shall be determined as follows:

1. Use narrow-range pH paper.
2. Do not contaminate the sample or subsample by contact with pH paper or pH electrode.
3. Use non-contaminating transfer implements, if necessary, to obtain a sample portion for use in the pH check procedure.
4. Check pH of VOC samples after taking aliquot for analysis, or check pH on duplicate sample that can be destroyed for this purpose
5. Pour a portion of the sample on the pH paper, unless the sample is an analytical portion that cannot suffer significant quantitative loss. In this case, transfer a test specimen with disposable pipet or other implement to the pH paper (see 5.4.8.3.c.3 above)

5.4.9 Tracking for Interlaboratory Transfer of Samples/Subsamples

If samples or sample extracts/digestates are sent to another laboratory, the information transmitted to the receiving laboratory must include, at a minimum:

1. Field ID number (optional)
2. Date and time of sample collection
3. Intended analyses by approved method designation
4. Method of preservation
5. Comments about sample or sample container (if applicable)
6. Date of sample preparation (if applicable)
7. Laboratory ID number (if applicable)

5.4.10 Sample Analyses Documentation Requirements

All sample analyses shall be completely documented by retaining all associated records. These records shall include, but are not limited to the following:

5.4.10.1 Information concerning all sample data:

- a. all sample identifications
- b. dates of analyses
- c. instrumentation ID and instrumentation parameters affecting the analytical run
- d. approved method numbers for the analyses performed

- e. all raw and reduced analytical data
- f. all calculations
- g. analyst's initials or signature

5.4.10.2 GC/MS analyses:

- a. Retain all electronically generated records (including the tune file and calibration date) on a write-protected diskette or tape in an orderly, logical manner; OR
- b. Retain the hard copy records of all data in the analytical run (blanks, QC samples, standards, samples, etc.) which must include:
 - 1. A copy of the total ion chromatogram, normalized to the highest non-solvent base peak;
 - 2. Complete quantitation report;
 - 3. Confirmation of all hits (mass spectra from the sample and library); and
 - 4. Mass spectra from all unidentified compounds that exceed 5% of the highest base peak (excluding solvent fronts). This includes retention time, tabulation of mass abundances, and mass spectra of the 5 most probable library hits.

5.4.10.3 Assure that all analysis data is linked with records for ancillary data and procedures (e.g. sample preparation).

5.4.11 Documentation Requirements for Other Laboratory Operations

The following activities, which are not specifically discussed in this Section, shall be documented according to the requirements found in the cited sections.

- 1. Preparation of Reagents and Analyte-Free Water - Section 6.2
- 2. Preparation of Analytical Calibration Standards - Section 7.2
- 3. Analytical Calibrations and Standardizations - Section 7.5 and 7.8
- 4. Preventative Maintenance - Section 8.0
- 5. Quality Control - Section 9.4
- 6. Corrective Actions - Section 11.6

5.5 ELECTRONIC DATA DOCUMENTATION

These requirements apply to all laboratory and field records which are generated or stored electronically.

5.5.1 Retention of Automatic Data Recording Products

1. All products or outputs of automatic data recording devices, such as chart strip recorders, integrators and computers, shall be retained either in electronic, magnetic or paper form.

2. All such records shall be properly identified as to purpose, analysis date, and field and/or lab ID number. The information in Section 5.4.10.1 shall be recorded for all laboratory and all applicable field analyses.

5.5.2 Electronic Data Security

1. Controlled or secured access to levels of data-editing capability are recommended. Software should provide prompts to the user for double-checking entries before executing deletions or changes to data. User-interaction or data-alteration tracking software is recommended, if available.

2. Raw data that is electronically collected from instrumentation shall not be altered in any fashion. Software that allows an analyst to correct raw data (e.g. change baseline) is acceptable.

5.5.3 Electronic Data Storage and Documentation

1. Electronically or magnetically stored data shall be easily retrievable for printing to paper.

2. All electronic/magnetic data files shall be coded, indexed, cross-referenced, etc., to allow linkage to sample data, analytical events and other laboratory procedural records. These file designations shall allow easy retrieval of the record.

3. All software algorithms employed to perform calculations required by the approved methodology or procedures shall be verified for accuracy and conformance with the methodology protocols, formulas, etc. This verification shall be documented.

a. This requirement applies to all automatic calculations and automatic data collection affecting calibrations, analyses, QC determinations, spread sheets, etc.

b. The vendor literature for software products may fulfill this requirement, if sufficiently detailed.

4. All software problems and their resolution shall be documented in detail, where these problems affect the correctness of laboratory data documented per this Custody SOP or where problems affect the cross-indexing of records. Record the calendar date, time, responsible personnel and relevant technical details of all affected data and software files. Indicate which files have been affected. All software changes, updates, installations, etc. shall be similarly documented per the above concerns. File and link all associated service records supplied by vendors or other service personnel.

5.6 LEGAL OR EVIDENTIARY CUSTODY PROCEDURES

The use of Legal Chain-of-Custody (COC) protocols are not required by DER. The following procedures are designed to document and track all time periods and the PHYSICAL POSSESSION AND/OR STORAGE of sample containers and samples from point of origin through the final analytical result and sample disposal.

This type of documentation is useful in establishing the evidentiary integrity of samples and/or sample containers. It can be used to demonstrate that the samples and/or sample containers were handled and transferred in such a manner to eliminate possible tampering. As such, these protocols are advantageous if data is to be used in legal cases such as law suits, criminal actions, enforcement actions, etc.

In addition to the records listed in Sections 5.1 through 5.5, the following protocols shall be incorporated IF legal COC is implemented by the organization:

5.6.1 General Requirements

1. The Chain of Custody records shall establish an intact, contiguous record of the physical possession, storage and disposal of sample containers; collected samples; sample aliquots; and sample extracts or digestates. For ease of discussion, the above-mentioned items shall be referred to as "samples":

a. The COC records shall account for all time periods associated with the samples.

b. The COC records shall include signatures of all individuals who were actively involved with physically handling the samples.

1. The signature of any individual on any record that is designated as part of the Chain of Custody is their assertion that they personally handled or processed the samples identified on the record.

2. Each signature shall be accompanied by a short statement which describes the activity of the signatory (i.e. received by, relinquished by, etc.).

c. In order to simplify record-keeping, the number of people who physically handle the sample should be minimized.

d. The COC records are not limited to a single form or document. However, organizations should attempt to limit the number of documents that would be required to establish COC.

1. DER recommends grouping activities on documents (e.g., a sample transmittal form to document field

- activities and laboratory receipt; a sample storage and disposal form to document storage; etc.).
2. A COC Form shall document all sample transmittals from one party to another (see 5.6.3).
 3. The laboratory records such as initial sample log records, sample preparation logs, analyst's run logs, etc. shall also be considered as part of the chain of custody unless the organization has established other records or protocols to document these laboratory functions.
2. Legal chain of custody shall begin when the precleaned sample containers are dispatched to the field.
 - a. A COC form must be signed by the person relinquishing the prepared sample kits or containers and by the individual who receives the sample kits or containers.
 - b. Thereafter, all parties handling the sample are responsible for sample custody (i.e. relinquishing and receiving) and documentation EXCEPT when the samples or sampling kits are relinquished to a common carrier.
 3. The common carrier should not sign COC forms.
 - a. The COC form shall indicate the name of a common carrier, when used. The shipping bill or other documents must be retained
 - b. All other transferor and transferee signatures associated with common carrier transfers are required. This shall include laboratory, field and other personnel releasing or accepting materials from the common carrier.
 - c. COC will be relinquished by the party who seals the shipping container and accepted by the party who opens it. The COC form shall indicate the date and time that the transport container was sealed for shipment.
 - d. Transport containers shall be sealed with strapping tape and a tamper proof custody seal. The custody seal must have space for the signature of the person who affixed the seal along with the date and time.
 4. The COC forms shall remain with the samples during transport or shipment. They must be put in a waterproof closure inside the sealed cooler or shipping chest.

5.6.2 Required Contents for Custody Records

Tracking records shall include, by direct entry or linkage to other records:

1. Time of day and calendar date of each transfer or handling procedure
2. Signatures of transferors and transferees
3. Location of samples (if stored in the field or laboratory)

4. Handling procedures (e.g. sample preparation, sample analysis, etc.) performed on the samples
5. Storage conditions for the samples, including chemical and thermal preservation
6. Unique identification for all samples
7. History of access to samples by all personnel, with personnel names recorded
8. Final disposition of physical sample
9. Common carrier documents

5.6.3 Required Information to be Included on COC Forms Used for Sample Transmittal

A Chain-of-Custody record or form shall accompany all evidentiary samples and subsamples that are transmitted and received by any party. The COC record or form shall specifically contain the following information:

1. Sampling site name and address
2. Date and time of sample collection
3. Unique field ID code for each sample source and container
4. Name of personnel collecting samples
5. Signatures of all transferors and transferees
6. Time of day and calendar date of all custody transfers
7. Clear indication of number of sample containers
8. Required analyses (by approved method number where applicable, see 5.3.4.1.f.)
9. Common carrier usage, if applicable (see 5.6.1.3)
10. Sample container/preservation kit documentation, if applicable (see 5.4 and 5.4.1.2), including kit ID #

5.6.4 Chain-of-Custody Seals

At a minimum, tamper-indicating tape or seals shall be affixed to all shipping container closures when transferring or shipping sample container kits, or samples to another party.

1. The seal shall be placed so that the transport container cannot be opened without breaking the seal.
2. The time, calendar date and signatures of responsible personnel affixing and breaking all seals shall be recorded on the seals.
3. Seals shall be retained as a part of the COC documentation.

[[4. While not required, organizations may elect to apply seals to individual containers. This establishes the history of each individual sample. The requirements specified for transport container seals shall be followed.]]

5.6.5 Controlled Access to Samples

1. Access to all evidentiary samples and subsamples shall be controlled and documented. The number of individuals who physically handle the samples should be limited to those responsible for sample collection, initial laboratory receipt, sample preparation and sample analysis (see 5.6.1.1.c) and sample disposal.

2. Samples and subsamples shall be placed in locked storage (e.g., locked vehicle, locked storeroom etc.) at all times when not in the possession or view of authorized personnel.

a. Some organizations maintain restricted access to their facilities and contend that storage under these conditions should constitute secure storage. This practice is acceptable as long as non-laboratory personnel (i.e. janitors, security guards, etc.) are not able to gain access to the samples after business hours.

b. Field personnel shall not leave samples in unoccupied motel or hotel rooms.

5.6.6 Transfer of Samples to Another Party

Transfer of samples, subsamples, digestates or extracts to another party are subject to all of the requirements of Section 5.6.

5.6.7 Sample Disposal

1. Disposal of the physical sample shall occur only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample.

2. All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.

3. Records shall indicate the date of disposal, the nature of disposal (i.e. sample depleted, sample flushed into sewer, sample returned to client, etc.), and the name of the individual who performed the task. Note: if samples are transferred to another party, custody transfer shall be documented in the same manner as other transfers (see 5.6.3 above).

6.0 ANALYTICAL PROCEDURES

6.1 LABORATORY GLASSWARE CLEANING PROCEDURES

In the analysis of samples the preparation of scrupulously clean glassware is mandatory. Lab glassware cleaning procedures must follow specific written method requirements. If procedures are not listed then the method of cleaning should be adapted to both the substances that are to be removed, and the determinations (tests) to be performed. Recommendations for such cleaning procedures are listed below.

If documentation through an active quality control program using spiked samples and reagent blanks can demonstrate that certain steps in the cleaning procedure are not required for routine samples, then those steps may be eliminated from the procedure.

Lab Glassware Cleaning Procedures

Analysis/Parameter	Cleaning Procedure (in order specified)

ORGANICS	
Semi-Volatile: (Pesticides, Herbicides, HPLC, Oil & Grease, TRPH & Total Recoverable Phenolics)	Solvents: 5, 1-4, 5 or 6, 13, 15 OR Muffle: 5, 1-4, 12, 13, 15 OR Oxidizer: 5, 1-3, 14, 3-5, 13, 15
Volatile or Purgeable (and EDB, DBCP, THMS)	1-4, (6 optional), 10 OR 1-4 (5 & 7 optional), 10
TOC, POX, TOX:	14, 1-4, 12
INORGANICS	
Trace Metals:	1-4, 9, 8 (optional), 4
Nutrients, Minerals:	1-4, 8, 4
Solids:	1-4, 11 (Volatile Solids 16)

Analysis/Parameter	Cleaning Procedure (in order specified)
Non-Metals, Physical Properties: (Cyanide, BOD, COD)	1-4, (14 optional BOD)
MICROBIOLOGY	1-4, (Sterilize per approved method)
BIOASSAY	
Freshwater:	18, 2, 3, 9 or 8, 4, 5, 4, 20
Marine & Estuarine:	19, 2, 3, 9 or 8, 4, 5, 4, 20
RADIONUCLIDES	17, 3, 8, 4

Cleaning Procedures:

1. Remove all labels using sponge or acetone.
2. Wash with hot tap water and a brush to scrub inside of glassware, stopcocks, and other small pieces, if possible, using a suitable laboratory-grade detergent.
 - Organics- Liquinox, Alconox or equivalents
 - Inorganic anions- Liquinox or equivalent
 - Inorganic cations- Liquinox, Acationox, Micro or equivalents
 - Microbiology- must pass inhibitory residue test
3. Rinse thoroughly with hot tap water.
4. Rinse thoroughly with deionized water.
5. Rinse thoroughly with pesticide grade Acetone.
6. Rinse thoroughly with pesticide grade Methanol.
7. Rinse thoroughly with pesticide grade Hexane.
8. Rinse or soak with 1:1 HCl (Hydrochloric Acid).
9. Rinse or soak with >10% HNO₃ (Nitric Acid).
10. Bake at 105 C for 1 hour.
11. Bake at 180 C (prior to use as per method).
12. Drain, then heat in muffle furnace for 30-60 minutes at 400 C.
13. Clean, dry glassware should be sealed and stored in dust-free environment.
14. Soak in oxidizing agent (Chromic acid or equivalent); preferably hot (40-50 C).
15. Last step (prior to use) should be a rinse with the solvent used in analysis.

Cleaning Procedures:

16. Drain, then heat in muffle furnace for 1 hour at 550 C.
17. Heat 1 hour in EDTA solution at 90-100 C.
18. New glassware must be soaked overnight in 10% HNO₃ or HCl.
19. New glassware must be soaked overnight in seawater.
20. Rinse thoroughly with dilution water.

CLASS A VOLUMETRIC GLASSWARE SHOULD NOT BE BAKED

6.2 LABORATORY REAGENT STORAGE

1. Laboratory reagents and chemicals must be stored according to method guidance and the manufacturer's instructions. All solvents used for VOC analyses shall be stored separately.

[[2. Reagents should be segregated according to compatibility groups(e.g. Solvents (flamm/nonflamm), bases, acids, reactive chemicals, etc.). Storage should follow all OSHA requirements.]]

3. A permanent record of reagent storage and preparation shall be maintained for all chemicals. At a minimum, these records shall document:

- a. storage conditions and location for reagents (implemented internal laboratory SOPs and/or safety plans that outline storage conditions and location may be used in lieu of specific reagent container records)
- b. vendor name
- c. date received/date opened
- d. expiration dates
- e. lot numbers
- f. preparation dates
- g. amounts and concentration of all source reagents and compounds used
- h. signature or initials of preparer.
- i. pH of microbiological culturing medias before and after sterilization

4. Documentation shall be maintained on all sources of analyte-free water. This documentation shall include records on all maintenance, cartridge-changing and miscellaneous tasks performed to upkeep or repair the system and all routine QC analysis protocols specifically scheduled and performed to monitor the system. Records must be maintained which identify the source and the specific use of analyte-free water that is obtained from commercial vendors.

6.3 LABORATORY WASTE DISPOSAL

Handling, storage and disposal of laboratory-related hazardous wastes are subject to the regulations contained in the Resource Conservation and Recovery Act.

It is the responsibility of the laboratory to store, package, label, ship and dispose of hazardous wastes in a manner which ensures compliance with all Federal, State and local laws, regulations and ordinances.

A waste is considered hazardous if:

1. The waste material is listed as hazardous in 40 CFR Part 261.30-261.33.
2. The material exhibits any of the characteristics of hazardous waste: (ignitability, corrosivity, reactivity or TC toxicity).
3. The waste is listed in 1 or 2 above and is not excluded by any provisions under the Resource Conservation and Recovery Act.

A waste is considered an acute hazardous waste if it is identified in 40 CFR Part 261.31, 261.32 or 261.33 (e) as an acute hazardous waste.

Laboratories that generate hazardous waste are put into 3 categories based on the amount of hazardous waste generated monthly. These categories are: 1) conditionally exempt small quantity generator; 2) small quantity generator and; 3) full generator:

1. Conditionally Exempt Small Quantity Generator

A generator who generates no more than 100 kilograms of hazardous waste or 1 kilogram of acute hazardous waste in a calendar month and accumulates no greater than 1000 kilograms of hazardous wastes (40 CFR Part 261.5).

2. Small Quantity Generator

A generator who generates 100-1000 kilograms of hazardous waste per calendar month and accumulates no greater than 6000 kilograms of hazardous waste or more than 1 kilogram per month of acute hazardous waste (40 CFR Part 262.34).

3. Full Generator

A generator who generates hazardous wastes in excess of 1000 kilograms per calendar month or more than 1 kilogram per month of acute hazardous waste (40 CFR Part 262.34).

It is the responsibility of the laboratory to know which category their organization falls under. Since most laboratories

will fall into the conditionally exempt small quantity generator category these disposal requirements are listed below.

Facilities falling into the small quantity generator and full generator categories must adhere to all regulations pertaining to waste, transport, storage and disposal in the Resource Conservation and Recovery Act.

Conditionally exempt small quantity generators must dispose of hazardous waste in an on-site facility or ensure delivery to a treatment, storage or disposal facility, which is:

1. Permitted under 40 CFR Part 270;
2. In interim status under 40 CFR Parts 270 & 265;
3. Authorized to manage hazardous waste by a state with a hazardous waste management program approved under Part 271; or
4. Permitted, licensed, or registered by a state to manage municipal or industrial solid waste*. *(subject to local regulations).

6.3.1 General Disposal/Treatment Considerations

1. Hazardous waste solvents, as identified in the 40 CFR Part 261 may not be evaporated off in a fume hood. Solvents evaporated off during the extraction/testing process are exempt.

2. Acidic & Basic wastes may be neutralized and disposed of via the sanitary sewer if they are not hazardous due to the presence of other constituents*. (*subject to local regulations).

3. Heavy metals may be precipitated out and the liquid portion disposed of via the sanitary sewer*. (*subject to local regulations).

6.3.2 Storage and Accumulation

Hazardous waste storage is limited to quantity and/or accumulation time and must comply with RCRA regulations as specified in the 40 CFR. These wastes should be packaged and separated according to compatible groups (e.g. solvents, acids, etc.).

6.3.3 Sample Disposal

Samples submitted to a laboratory for analysis are excluded from regulation as hazardous waste under 40 CFR Part 261.4(d) provided the samples are being transported to or from the laboratory, are being analyzed, are being held for analysis or are being maintained in custody for legal reasons. However, once

a decision is made to dispose of laboratory samples, the exclusion provisions of 40 CFR Part 261.4(d) no longer apply. Samples that have been identified as hazardous may either be: 1) returned to the generator; or 2) disposed of according to applicable RCRA regulations summarized in this document. Samples which are determined to be non-hazardous may be subject to local environmental regulations. It will be the responsibility of the laboratory to be familiar with any such local regulations.

A sample collector shipping samples to a laboratory and a laboratory returning samples to a sample collector must comply with U.S. Department of Transportation (DOT), U.S. Postal Service (USPS), or any other applicable shipping requirements.

7.0 CALIBRATION PROCEDURES AND FREQUENCY

7.1 INTRODUCTION

This SOP stipulates minimum calibration requirements necessary to ensure that the measuring system is capable of producing acceptable data. Acceptable calibration protocol must involve a demonstration that the instrument or measuring system is capable of acceptable performance at the beginning of the analysis sequence and that initial calibration is still valid after continued system operation.

7.2 GENERAL CONSIDERATIONS

7.2.1 Calibrations must be performed according to all analytical method directives OR as indicated in this Guidance Document if specifics are not addressed in the cited method.

7.2.2 Analytical method calibration acceptance criteria must be followed or if acceptance criteria are not specified in the method, general criteria presented in this SOP shall be used to verify an acceptable calibration.

7.2.3 The number of calibration standards used to achieve an acceptable calibration must adhere to the cited method. If this information is not in the method, then a minimum of a blank and 3 standards must be employed to develop calibration curves. See Section 7.6.3 for guidance on other types of analyses.

7.2.4 At least one of the calibration standards shall be at a concentration of 1 - 2 times the laboratory practical quantitation limit for the method. By using a calibration standard at that level, the laboratory can verify the PQL with each initial calibration.

7.3 STANDARD RECEIPT AND TRACEABILITY

7.3.1 Records to be retained for primary stock standards must include source, type of standard, date of receipt, lot number (if applicable), expiration date and purity statement.

7.3.2 Records to be maintained for preparation of intermediate standards must include identification of primary standards used, preparation date, methods of preparation (including specific dilution information), preparer identification, concentration prepared and expiration date.

7.3.3 Preparation records for working standards must include identification of primary and intermediate standards used in

working standard preparation, date of preparation, method of preparation (including dilutions), concentrations prepared and preparer identification.

7.4 FREQUENCY OF STANDARD PREPARATION AND STANDARD STORAGE

7.4.1 Standard Storage

1. Standards must be stored according to analytical method guidance or supplier recommendations.

2. If no method or supplier guidance is available standards must be replaced upon decreased instrument response.

7.4.2 Frequency of Standard Preparation

1. If no method or supplier guidance is available standards must be renewed upon decreased instrument response.

2. It is recommended that all primary standards be held for no longer than one year.

3. Working standards are to be prepared on a daily basis unless specific method guidance stipulates differently.

7.4.3 Tables specifying standard storage protocol and standard preparation frequencies must be available for inspection at the laboratory.

7.5 MINIMUM CALIBRATION REQUIREMENTS FOR FIELD INSTRUMENTS

This section will discuss pre-inspection calibration, field calibration, office/lab calibration, and use of field instruments. Please note that instrument-specific or model-specific calibration and operation procedures are not included. If the following procedures do not apply to your particular equipment, the pertinent analytical reference and the manufacturer's operating/owner's manual shall be used for specific protocols.

7.5.1 General Considerations

7.5.1.1 Calibration of field instruments shall be performed on a regular basis with records kept on the field sheets, field logs or in a separate calibration log. The records must indicate the method used to calibrate, the time and date, number of standard(s), resulting meter response, actions taken, and the results of the calibration. Optionally, the meter: name, model number, and identification number (if applicable) may be entered.

7.5.1.2 Maintenance and repair notes shall be made in the maintenance logbook for each meter. If rental equipment is used, a log is not required. However, the origin (i.e. rental company), rental date, equipment type, model number and identification number (if applicable) shall be entered into the field notes or a rental equipment notebook

7.5.1.3 Prior to mobilization, the Project Manager must verify that all equipment is in proper working condition, calibrated, and that batteries are properly charged.

7.5.1.4 Field calibration of each meter shall occur daily, at the first sample site and must be verified throughout the day (see 7.5.1.5 below). This will ensure field data of a known quality. All field calibrations and checks shall be noted on field sheets.

7.5.1.5 Minimum Quality Control Requirements

a. The QA Rules no longer require the generation of historically derived QA Targets of precision and accuracy for field measurements. In lieu of taking duplicate measurements and using independent QC check standards, more frequent continuing calibrations shall be performed.

b. Once the meter has been calibrated, these checks shall take place at intervals of no more than 4 hours and at the end of the sampling day. For instance: the pH meter will be checked against the pH 7 buffer, thermistors will be checked against field-grade thermometers, conductance meters will be checked against one KCl standard, etc.

c. If a field meter fails a continuing calibration, a complete initial calibration must be performed. In this way, meter response will be addressed without the need for generating historical precision and accuracy statistics.

7.5.1.6 Documentation on calibration standards (e.g., buffers, KCl, and other reagents) must be maintained.

a. At a minimum, the date of receipt, expiration dates (noted on the bottle label), and date of first use shall be noted on the standard container.

b. Expiration dates must be followed.

c. If reagents or standards are prepared from stock chemicals, they must be analytical reagent grade or better.

NOTE: Potassium chloride standards must be of primary standard grade.

7.5.2 pH Meters:

7.5.2.1 General Concerns:

a. The pH meter is field calibrated on a daily basis at the first site. Since field meters do bump around from site to site, calibration is likely to change. Calibration checks must be made per 7.5.1.5 above.

b. Calibration may be checked on a weekly basis in the office or laboratory to ensure the % theoretical slope is not less than 90%, indicating a bad electrode. This should be noted in the calibration records. If % slope cannot be determined on your meter, or the manufacturer's optimum specifications are different, manufacturers recommendation for maintaining optimum meter performance shall be followed.

c. There are several interferences to keep in mind with pH measurement:

1. sodium at pH > or = 10 can be reduced or eliminated by using a low sodium error electrode;

2. coatings of oils, greases, and particulates may impair the electrode's response. The electrode bulb should be patted dry with lint-free paper or cloth and rinsed with deionized water. If not, acetone may be used to clean very hard to remove films, but must be used sparingly so the electrode surface is not damaged;

3. temperature effects on the electrometric measurement of pH are controlled by using instruments having temperature compensation or by calibrating the meter at the temperature of the samples;

4. poorly buffered solutions with low specific conductance (<200 umhos/cm) may cause fluctuations in the pH readings. Equilibrate electrode by immersing in several aliquots of sample before taking pH.

d. Follow the instructions with each type of pH meter. Use secondary standard buffer solutions (pH of 4, 7, 10) purchased from commercial vendors for calibration. Do not reuse buffers.

e. Each meter/electrode system must be calibrated at a minimum of two points, at least three pH units apart, bracketing the expected sample pH. Check historical data for expected pH or use pH paper on an aliquot to estimate.

f. Under normal conditions a pH measurement should be accurate to +/- 0.1 pH unit. Remember the needle of the pH

meter must align with its image on the mirror on the gauge to get an accurate reading. Similar care must be taken when recording digital read-out.

7.5.2.2 Calibration and Field Use

- a. Check the battery before mobilizing and turn on the meter when you reach the first facility and allow it to equilibrate to ambient temperature.
- b. Calibrate the meters prior to taking samples:
 1. Estimate the sample pH range (e.g., history, operator, litmus)
 2. Turn function switch to pH position
 3. Select the appropriate buffers to bracket the expected sample pH, either pH 4 buffer and pH 7 or pH 7 and pH 10.
 4. Remove the protective cap, rinse the electrode with deionized water (DI) and dab dry with lint-free paper or cloth.
 5. Place and swirl the electrode in the pH 7 buffer and turn the calibration knob until the readint is 7.0. Repeat step 4 above.
 6. Place and swirl the electrode in the second buffer solution (pH 4 or 10). Adjust the temperature knob until until the reading is that of the pH standard. Repeat step 4 above.
 7. Measure the temperature of the second buffer solution.
 8. Turn the slope indicator until the arrow of the temperature compensator points to the temperature of the buffer. The percent to the theoretical slope should be read from the slope scale. A slope of less than 90% (or one not meeting the manufacturer's specifications) indicates a faulty electrode or contaminated buffer and the problem should be corrected before proceeding.
- c. After calibration follow these procedures to take a pH reading of a freshly collected sample:
 1. Pour enough fresh sample into a pH measuring cup to take a reading and measure its temperature. If it differs more than 2 C from the buffer temperature, adjust for the difference by turning the slope indicator until the arrow to the temperature compensator points to the sample's temperature.
 2. Place and swirl the pH electrode in the sample (in the cup) and read the pH value. In the case of low specific conductance and meter drift, add 1 ml of 1M KCl (potassium chloride) solution to each 100 ml of sample, swirl and read pH. Note: to make 1M KCl solution, take

74.55 grams of primary standard grade KCl and add it to a 1 liter volumetric flask. Add DI to the 1 liter line on the flask and mix. Solutions of the appropriate strength may be purchased from commercial laboratory suppliers.

3. Turn the meter off after the last reading, discard the sample in the cup, rinse the electrode thoroughly with deionized water and replace the electrode's rubber cap.

d. The QAS no longer requires performing duplicate measurements (precision) or independent check standards (accuracy). These QC checks are optional and do provide an excellent check of instrument response and operation. In lieu of performing these checks, additional calibration checks will be mandatory. Continuing calibration must be done per the following:

1. After the initial calibration, the pH meter shall be checked against the pH 7 buffer at intervals of no more than 4 hours.

2. The meter will also be checked against the 7 buffer after sampling has been completed.

3. If the sampling event takes less than 4 hours, then an initial calibration and a post-calibration check will be adequate.

4. If, during the continuing calibration, the response is greater than .2 pH units on either side of 7, then a complete initial calibration must be conducted.

5. All initial and continuing calibrations shall be completely documented in bound notebook or field sheets, including: date/time, standard(s) used, resultant meter response, action taken, and technician initials.

7.5.3 Temperature

7.5.3.1 General Concerns

a. Temperature determinations can be made with any field-grade mercury-filled, alcohol-filled, or dial-type Celsius thermometer as well as an electronic thermistor. The dial type thermometer is preferred over the glass type for field work because of its durability and ease of reading.

b. All thermometric devices shall, at a minimum, be checked annually in the laboratory against a National Institute of Standards and Technology (NIST) precision thermometer. If data is generated for submission to DER as a Monthly Operating Report for domestic or industrial wastewater, this calibration check must be increased to quarterly.

1. The temperature measuring device should be checked at two temperatures against the NIST precision thermometer.
2. Temperatures should agree within ± 0.1 C. Make note of the calibration in the calibration records. Note the make, model, and serial number of each thermometer.
 - a. Thermometers that do not meet the acceptance criteria should be disposed of properly.
 - b. If the difference is shown to be constant (i.e. ± 0.5 C) over the thermometer range, the thermometer may be used provided that the difference is documented for 10 degree increments, and the correcting factor is used in all measurements.
- c. Use care and proper cleaning procedures to prevent sample cross-contamination.

7.5.3.2 Calibration and Field Use

- a. All field-grade thermometers must have completed the annual check against the NIST-grade thermometer. All thermistors must be calibrated in the field with a field-grade (or NIST-grade) thermometer.
- b. Allow the thermometer or thermistor (always use one which has been properly calibrated) to equilibrate to ambient temperature.
- c. Insert thermometer or thermistor in situ when possible or in a portion of the sample. Swirl and take readings when the mercury column, needle, or read-out becomes constant; record the temperature to the nearest 0.5 C. Read to the nearest 0.1 C for a digital gage.
- d. Continuing calibration must also be performed for thermistors. The thermistor should be checked against the field-grade thermometer at 4 hour intervals and at the end of the sampling day.

7.5.4 Dissolved Oxygen Meter

7.5.4.1 Introduction

The electrode method is predominantly used in situ for dissolved oxygen (DO) determinations.

7.5.4.2 General Concerns

- a. Before sampling the DO meter should be calibrated in water saturated air to make sure it is operating correctly. The DO meter should be calibrated on samples free of

interference, in the laboratory, and against the Azide modification of the Winkler Method of determining dissolved oxygen on an annual basis.

b. Turbulence is necessary to keep a constant flow of water across the membrane-sample interface. Be sure the stirrer is working before using the probe.

c. Store the probe with a cover that creates a saturated atmosphere. A cap, with a wet sponge in it, will suffice.

d. Before mobilizing, check to make sure there are no bubbles beneath the probe membrane and no wrinkles or tears in the probe membrane. If so, replace the membrane and KCl. Check the leads, contacts, etc. for corrosion and/or shorts if meter pointer remains off-scale, does not calibrate, or drifts.

e. Dissolved inorganic salts are an interference with the performance of DO probes. For example, the taking of DO readings in salt water is affected by the salinity and must be corrected by adjusting the salinity knob. Adjust the meter based on readings taken from the specific conductivity/salinity meter or use appropriate calculations to correct for salinity.

f. Reactive gases which pass through the membrane may interfere. For example, chlorine will depolarize the cathode and cause a high probe output. Long term exposures to chlorine will coat the anode with the chloride of the anode metal and eventually desensitize the probe. Sulfide (from H₂S) will undergo oxidation if high enough potential (voltage) is applied, creating current flow, yielding faulty readings. If such interferences are suspected, the membrane electrode should be changed frequently, and must be calibrated at more frequent intervals.

g. DO probes are temperature sensitive, and a method of temperature compensation is normally provided by the manufacturer.

7.5.4.3 Calibration and Field Use:

a. Annual Laboratory Calibration

1. Fill a clean bucket with uncontaminated or deionized water and place the probe into the bucket. Siphon water from the bucket into two Biological Oxygen Demand (BOD) bottles. Make sure to place siphon hose on the bottom of the bottles and overflow the bottles by three volumes.

Determine the DO by the Winkler method (see Standard Methods for the Examination of Water and Wastewater for more details).

2. Adjust the DO meter according to manufacturer's instructions. Be sure to adjust the meter to the temperature of water in the bucket, then calibrate the DO indicator dial to read the average DO concentration of the two samples determined by the Winkler test.

3. Keep a calibration log.

4. If the air calibration seems to operate properly but the oxygen concentrations disagree with the results of the Winkler calibration by more than 0.2 mg/L it is time to have the electrode or meter serviced or replaced.

b. Prior to mobilizing and at each sample site, air calibrate the DO meter in water saturated atmosphere to make sure the meter is reading correctly.

1. Turn meter on for at least 10 minutes before the initial field calibration and use. With lint-free paper or cloth, wipe any droplets off the membrane surface. For YSI meters, and most others, the meter must remain on redline to keep the membrane polarized. Do not turn off until the end of the day.

2. Once the probe/calibration chamber are stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table (usually on the meter's battery pack), what the DO should measure. (You can't get a stable ambient temperature if the probe is sitting in the sun).

3. With the probe as close to the water surface as possible (saturated atmosphere) turn the knob to read DO. Adjust the calibration knob until the DO reading is at the theoretical level determined in b.2. above.

c. Using the salinity measurement (if appropriate) from the conductivity meter, adjust the salinity control knob on the DO meter (ignore if your meter automatically adjusts for salinity). Take the DO reading and record it on the field sheet.

d. Place the DO probe at the depth and location appropriate to what you are measuring. For example, take the DO of an effluent just before it enters a receiving water. If the effluent has cascading or other aeration prior to entering the surface water, take the DO reading in the receiving water right where it enters. For well mixed surface waters, e.g., fast flowing streams, take the DO reading at approximately 1-2 feet below the surface or at mid-depth. For still or sluggish surface waters, take a

reading at one foot below the surface, one foot above the bottom, and at mid-depth. If it is shallow, say less than two feet, take the reading at mid-depth. Do not take a reading in frothy/aerated water since you may get a false reading.

e. Keep the probe in the saturated atmosphere (see 7.5.4.2.c above) between sites and events. If the readings show distinct, unexplainable changes in DO levels, or when the probe has been in waters with high sulfides, recalibrate using the Winkler method.

f. While taking a reading, if it is very low, e.g., below 1.0 ppm, allow it to stabilize, record it and then, remove and rinse the probe, as the environment is very likely anoxic and may contain hydrogen sulfide, which can damage the probe.

g. Continuing calibration must also be performed on the DO meter. The meter should be air calibrated at 4 hour intervals and at the end of the sampling day.

7.5.5 Specific Conductivity Meter

Specific conductance is a useful method to approximate the total amount of inorganic dissolved solids. Conventional conductivity devices consist of two or more platinum electrodes separated by a test solution. The major disadvantage with this type of system is the possibility of polarization or poisoning (fouling) of the electrodes. Conductivity systems based on the measurement of inductance or capacitance are also available. The electrodes in these systems are insulated by a layer of glass or other insulating material. System response is less rapid, but problems with fouling and polarization are eliminated. Conductivity varies with temperature. For example, the conductivity of salt water increases 3%/degree C at 0 C, and only 2 %/degree C increase at 25 C. Therefore, it is necessary to record temperature with conductivity measurements or to adjust the temperature of the samples prior to making conductivity measurements. Most conductivity meters have temperature compensation.

7.5.5.1 General Concerns

- a. Follow the manufacturer's instructions.
- b. Samples are preferably analyzed at 25 C. If not, temperature corrections are made and results reported at 25 C.
- c. With good equipment an accuracy of +/- 1% of the reading is achievable.

d. Typically a conductivity meter is combined with a thermistor to measure water temperature. The temperature measurements are used for both conductivity and DO corrections.

7.5.5.2 Calibration and Field Use

a. The meter should be checked in a laboratory in one of three ways:

1. Follow method specifications;
2. Use two standard potassium chloride solutions of 100 and 1,000 umhos/cm or standards that bracket the range of expected sample conductance; or
3. A single check standard in each range of a multi-range instrument.

b. If the meter does not read within 1% of the standards, determine what the problem is and correct it before proceeding. Most field instruments read conductivity directly. If the meter does not correct all values to 25 C, calculate corrective factors using the procedure in 7.5.5.3 below. Record all readings and calculations in the calibration records.

c. The meter must be calibrated in the field with at least one KCl standard prior to analyzing the first sample. The chosen standard must be close to the conductance value of the real samples.

d. Use during a sampling event:

1. Turn the meter knob to redline before use. Follow the manufacturer's recommendations or redline approximately 15 - 20 minutes before use.
2. When at a site or facility adjust the redline knob to align the needle directly over the redline, using the mirror reflection, if available.
3. Typically, the conductivity probe is immersed at the same time, depth, and location as the DO probe. Measure the water temperature with the conductivity probe.
4. If the meter is equipped with automatic temperature compensation, adjust the temperature knob on the conductivity meter to the water temperature and read the conductivity. The conductivity meter has a set of positions which multiply the reading by powers of ten in order to measure the full range of potential conductivities. You will need to set this dial to the correct range in order to take a reading. The reading, with the temperature gauge adjusted properly, reports conductivity measured at 25 C.

5. Switch the dial to take a salinity reading. Use this reading to adjust the DO meter for salinity, if necessary. This should not be used for reporting salinity as a measured parameter, since the calibration is not directly applicable. It may be used as an estimate for salinity for compensation of a DO measurement.

6. If using at more than one site or sampling location, keep the probe polarized by turning the meter's knob to redline and keeping the probe in water between locations.

7. Continuing calibration must be performed on the conductance meter. The meter should be checked against the one KCl calibration standard at 4 hour intervals and at the end of the sampling day.

8. Rinse off the probe with deionized water and turn off when finished for the day. Store the probe in deionized water at all times, if it dries out it takes 12 - 24 hours to rejuvenate it.

7.5.5.3 Calculations

a. If the meter does not automatically correct for temperature, or if a probe with a cell constant other than 1 is used, the following formula shall be used to correct the data to 25 C:

$$K = \frac{(K_m)(C)}{1 + 0.0191(T-25)}$$

Where: K = conductivity in umhos/cm at 25 C

K_m = measured conductivity in umhos/cm at T degrees C

C = cell constant

T = measured temperature of the sample in degrees C

If the cell constant is 1, the formula for determining conductivity becomes:

$$K = \frac{(K_m)}{1 + 0.0191(T-25)}$$

b. Refer to SM 2510B, 17th edition, if other calculations (i.e. determining cell constant, etc.) are required.

7.5.6 Chlorine Measurements

Residual chlorine is unstable in aqueous solutions and as such its concentration decreases rapidly with time. Exposure to sunlight (or other strong light) or agitation will accelerate chlorine reduction; therefore, analysis should begin immediately after sampling. Field colorimetric kits are available to test for the presence of chlorine. The colorimetric method which requires the use of a spectrophotometer (HACH DR-100) and the

amperometric method are approved by EPA. Some visual colorimetric tests using DPD chemistry and color wheels are EPA approved for domestic wastewater sources. The colorimetric spectrophotometer method is more desirable because of its ability to be calibrated. The subjective nature of assessing a titration endpoint used in the amperometric method reduces precision. The colorimetric method reduces human error. The amperometric method is better to use when there are matrix interferences in the wastewaters. For example, the lignins in pulp and paper wastewaters could cause a background color interference in the colorimetric method. A complete discussion of the methods are found in Standard Methods for the Examination of Water & Wastewater, 17th. Edition.

7.5.7 Organic Vapor Meters

Organic vapor meters may be used to perform qualitative or screening procedures in many different situations. These devices are equipped with either a flame ionization (FID) or a photoionization (PID) detector. The FID ionizes organic molecules via a hydrogen flame, whereas the PID uses a lamp. Lamps with different electron voltage (eV) may be used with the PID to ionize specific groups or classes of organic compounds. For specific lamp applications consult the owners manual.

These meters may be used for ambient air screening at sites for health and/or safety reasons. They can be used for headspace analyses of soil samples to determine "gross contamination" (17-770 F.A.C.), for well placement, or for grid sampling. Calibration and use of these types of meters should be performed after consulting the owners manual. There are several procedures that must be accomplished at a minimum:

1. Calibration must be performed on-site, prior to sampling, it is also suggested that additional calibrations against one span gas be performed at 4 hour intervals and/or at the end of the sampling day.
2. The meter must be zeroed with "zero air" or equivalent. If known to be free from interfering components, ambient air may be used.
3. At least one span gas must be used for calibration.
4. Carbon filters must be used to distinguish between methane and other aliphatic halocarbons (FIDs only).
5. Background corrections must be made if soil borings or split spoon samples are analyzed in ambient air (unnecessary for headspace samples performed in mason jars under foil).

6. Meters with PIDs must be calibrated against a meter with a FID if headspace samples are being performed for assessing "gross contamination" as defined in the Tanks rule, Chapter 17-770, FAC.

7.5.8 Automatic Wastewater Samplers

These pieces of equipment are invaluable for remote sampling or for sophisticated time- or flow-dependent sampling regimes. Since loading calculations of industrial and domestic wastewater are dependent upon the sampling accuracy, these devices must be volume calibrated by checking the constant pumping volume at least twice with a graduated cylinder or other calibrated container.

7.6 LABORATORY INSTRUMENTS

7.6.1 Initial Calibration

1. Instruments must be initially calibrated each time the instrument is set up or upon failure of any quality control calibration checks.

2. The number of standards to be used for initial calibration must conform to method protocol or general requirements in Section 7.6.3.

3. Correlation coefficients for photometric analyses must be calculated and documented and should be greater than or equal to 0.995.

4. A minimum of one quality control check standard at a mid-range concentration shall be analyzed prior to sample analyses to verify initial calibration. This quality control check standard shall be prepared independently of the calibration standards. Recoveries for this check standard should be between 90 and 110%, or as specified by the method.

7.6.2 Continuing Calibration

1. One mid-range continuing calibration standard must be analyzed for each group of 20 samples analyzed. The check standard used for initial calibration verification will verify acceptable calibration for the first set of 20 samples. Subsequent sample sets of 20 or portions thereof (if a complete set of 20 is not available), must have a continuing calibration check standard analyzed at the beginning of each sample set.

2. Recovery for the continuing calibration check standard shall be between 80 and 120%, the range specified by the analytical method or the documented acceptance range that is determined by internal historical data (see 9.2.3.4).

7.6.3 General Calibration Recommendations by Specific Analysis or Analysis Type**

1. Titrimetric Analyses - Standardize all titrants just prior to use.
2. Residue or Solids Analyses
 - a. Analyze Quality Control Check Samples on a quarterly basis.***
 - b. See calibration requirements for analytical balances and ovens (Section 7.7.1 and 7.7.3).
3. Conductivity
 - a. A minimum of 2 KCL standards must be analyzed bracketing the expected concentration of the samples to be analyzed.
 - b. The readings for the calibration standards must be within 1% of the expected value.
 - c. Continuing calibration checks must be within 1% of the true value.
4. Turbidity
 - a. Calibration must be checked for each instrument testing range applicable to the levels of turbidity to be measured.
 - b. If formazin standards are not used for the daily calibrations, then formazin standards must be prepared on a quarterly basis and compared with daily standards.
 - c. Calibration must be checked every 20 samples with 1 standard in each applicable testing range.
 - d. Acceptance criteria for all calibration and standard checks must be established per instrument accuracy specifications.
5. Dissolved Oxygen
 - a. Probe - Calibrate against Winkler Titration on an annual basis. Results should agree within 0.2 mg/l.
 - b. Winkler Titration - see titration section (7.6.3.1).
6. Color and Chlorine
 - Final determination made by comparison against Nessler Tubes or sealed color standards.
 - a. Confirm results against an approved alternate test procedure on a quarterly basis.
 - b. Results should be within 10% of the original value.

7. Temperature
 - a. Laboratory thermometers must be checked against an NIST certified thermometer on an annual basis. Results must be within the manufacturer's specifications.
 - b. Other devices used to record temperature must be checked on a monthly basis against a thermometer that has been calibrated against an NIST certified thermometer.
8. BOD
 - a. Analyze a glucose/glutamic acid check sample each day BODs are analyzed.
 - b. Check standard recovery must satisfy method criteria.
 - c. See Dissolved Oxygen calibration protocols (7.5.4).
9. Oil and Grease
 - a. See calibration criteria for the analytical balance (7.7.3).
 - b. Analyze a QC check sample on a quarterly basis (all applicable matrices).
10. Flash Point
 - a. Analyze a solution of known flash point each day of operation.
 - b. The flash point temperature should be within 5% of the literature flash point value.
11. Salinity
 - a. Electrical Conductivity Method - follow protocols for conductivity calibration and standardize instrument for seawater analyses according to method protocol on a semiannual basis.
 - b. Argentometric Method - standardize titrant daily and check method against a known seawater sample or alternate method quarterly.
 - c. Hydrometric Method - check method against the argentometric method or with a QC check sample quarterly.
 - d. Alternate method comparisons should agree within 10%.
12. Chlorophyll - analyze a QC check sample quarterly (if available).
13. Sulfate
 - a. Gravimetric - analyze a QC check sample quarterly and follow calibration requirements for the analytical balance (Section 7.7.3).
 - b. Turbidimetric - see requirements for calibration of turbidity (Section 7.6.3.4).
 - c. If sulfuric acid is used for standard preparation, then it must be standardized with each preparation.

7.7 SUPPORT EQUIPMENT CALIBRATION

7.7.1 Temperature Monitoring

1. Ovens - temperature recorded daily. Temperatures must be within acceptable method range.
2. Incubators and water baths - monitor temperature twice daily for microbiological work and once for other applications. Temperatures must be within acceptable method ranges.

7.7.2 Autoclaves - must document that sterilization temperature and pressure has been achieved by the use of sterilization indicators with every autoclave run.

7.7.3 Analytical Balances - monthly monitoring of Class S Weights. Results must fall within the suppliers acceptance criteria.

7.8 CALIBRATION DOCUMENTATION

Records must be maintained to document and verify acceptable instrument or measuring system calibration for each analysis.

7.8.1 Records must be maintained for all standard preparations and working standards must be easily traced to intermediate and primary standards used for preparation.

7.8.2 Acceptable calibration verification (% recoveries, correlation coefficients) must be recorded and easily identified with applicable daily calibrations.

7.8.3 If calibration acceptance criteria are based on manufacturer's instrument specifications or acceptable recoveries specified by QC check sample suppliers, then records of such activities must be maintained. Such records must be easily accessible and must establish verification of acceptance criteria.

7.8.4 Laboratories must have available for inspection a table specifying calibration acceptance criteria for all parameters.

7.9 DEFINITIONS

7.9.1 Mid-Range Standard - a standard in the middle of the linear range of the established calibration curve or a standard concentration in the middle of the expected sample concentration range depending on the type of determination to be performed.

7.9.2 Intermediate Standard - a standard prepared from the primary stock standard which is diluted to prepare the working calibration standards.

7.9.3 Working Standards - the standards that are actually analyzed to perform the instrument or measuring system calibration.

* Acceptance criteria presented in this guidance document are general advisory limits. Variances to the listed criteria must be supported with documentation. If the method stipulates different criteria, then the method criteria must be used to verify acceptable calibration.

** If analysis or analysis type is not mentioned in this SOP then method calibration protocol and general requirements as presented in this guidance document must be followed.

*** Recoveries for QC Check Samples should be between 90 and 110% or within acceptable ranges specified by the supplier.

8.0 PREVENTIVE MAINTENANCE

Preventive maintenance is the key ingredient to possessing analytical equipment that will produce reliable data over the life of the instrument.

Responsibility for preventive maintenance lies with the analyst and supervisory personnel in charge of the monitoring equipment. The analytical staff must be dedicated to the implementation of the preventive maintenance program and always watchful for signs that there is a need for maintenance activities. The analyst and supervisory personnel must be supported by vendor specialists or in-house experts that handle activities beyond simple repairs or maintenance.

The Preventive Maintenance Program must consist of:

1. A written PM schedule;
2. Documentation of all maintenance and repairs (records must be kept in an easily accessible manner);
3. Vendor operation and maintenance manuals available for all instrumentation; and
4. A written contingency plan specifying that backup equipment will be maintained for all instrumentation or stating that sampling events will be postponed and current sample load be invalidated until repairs are accomplished. If samples are sent to another laboratory the subject laboratory must have an approved CompQAP for the parameters of concern and the DER Project Manager must be notified if the analytical work is being performed under a Quality Assurance Project Plan.

Table 8.1 identifies preventive maintenance activities by instrument type required by DER with recommended frequencies. Please note that it may be necessary to perform activities more frequently depending on heavy workloads, sample types analyzed and/or instrument performance. If the instrument manufacturer recommends more frequent or additional maintenance activities these shall also be incorporated into the facility maintenance program.

TABLE 8.1
 PREVENTIVE MAINTENANCE ACTIVITIES

INSTRUMENT/ACTIVITY	FREQUENCY
AA SPECTROPHOTOMETER (FLAME)	
Clean nebulizer	SA
Clean spectrophotometer quartz windows	W
Burner head cleaned; check tubing, pump and lamps	D(1)
O rings checked	M(1)
Fine Tune Wavelength; Check optics	A
Check electronics	A(3,4)
AA SPECTROPHOTOMETER (FURNACE)	
Check graphite tubes	D(1)
Flush autosampler tubing	D
Replace graphite electrodes and Shrouds	SA
Clean furnace housing and injector tip	D
Check electronics	A(3,4)
AA SPECTROPHOTOMETER (COLD VAPOR & HYDRIDE)	
Flush tubing (automated systems)	D(1)
Check absorption cell for vitrification	D(1)
Replace or clean quartz cell	3
Check electronics	A(3,4)
ICP	
Clean and realign torch	M
Clean nebulizer and spray chamber; Check peristaltic pump tubing and vacuum pump oil	W(1)
Check entire optical system (mirrors, windows, etc.)	A(3,4)
Check water lines, torch compartment and gases	D
Check electronics (voltages, waveforms, etc.)	SA
Check wavelength calibration and adjust as needed	SA
Run interference (interelement) standard	SA
GAS CHROMATOGRAPHS	
GENERAL	
Check septa, cylinder gas pressure, oxygen/moisture traps	D
Bake out injector body	2
Check electronics (voltages, waveforms, etc.)	Q(3,4)
Check GC temperature calibrations (injector, oven, detector)	Q
COLUMNS	
Change glass sleeve inserts, shorten ends of columns, change glass wool plugs, check for leaks or replace	3
ELECTRON CAPTURE DETECTOR	
Wipe Tests	SA

TABLE 8.1, continued
 PREVENTIVE MAINTENANCE ACTIVITIES

INSTRUMENT/ACTIVITY	FREQUENCY
ELECTRON CAPTURE DETECTOR (CONT'D)	
Hydrogen cleaning	3
Returned to factory for cleaning and refoil	3,4
FLAME IONIZATION DETECTOR	
Clean	Q
Replace Flame Tip	A
PHOTO IONIZATION DETECTOR	
Clean lamp	M(1)
HALL ELECTROLYTIC CONDUCTIVITY DETECTOR	
Replace resin, change solvent and clean conductivity cell	3,4
Change Ni tube	Q
NITROGEN PHOSPHORUS DETECTOR	
Clean	Q
MASS SPECTROMETER	
Replace vacuum pump oil and change desiccant	A
Check ion source and analyzer (dismantle and clean, replace parts as needed)	Q
Check mechanicals (vacuum pumps, relays, gas pressures and flows)	Q
Check mass calibration w/ FC-43 (perfluorotributyl-amine)	D
PURGE AND TRAP	
Clean sparger	W
Change Trap	A
Bake Trap	2
Check purge flow	M
Check for leaks	M
HIGH PRESSURE LIQUID CHROMATOGRAPHY	
Gas lines checked for leaks	D
Clean mobile phase flow system with nitric acid	SA
Clean detector flow cells with nitric acid	SA (3)
Clean injection valve	A
Check solvent filters	W
Check pumps seals and check valve assemblies (clean and replace as pressures & flows of mobile phase indicate)	D
Lubricate oil felts, if present	M
Lubricate post column reagent pumps and check valve assembly oil felts	M

TABLE 8.1, continued
 PREVENTIVE MAINTENANCE ACTIVITIES

INSTRUMENT/ACTIVITY	FREQUENCY
INFRARED SPECTROPHOTOMETER	
Clean instrument housing	M
Change desiccant and clean cells	Q
Clean windows	M
AUTOANALYZERS	
Check for leaks, flush out system and clean up spills after use	D
Clean sample probe and check all tubing for wear and discoloration	M(1)
Clean optics	Q
Oil sample motor, lubricate gears, clean flow cell	SA
Clean pump rollers, platens and colorimeter filters	M
ION CHROMATOGRAPH	
Check for leaks	D
Check all lines for wear and discoloration	W(1)
Check pump pistons	A
TOC ANALYZER	
Change injection needle, clean injection port, and change catalyst	M
Inspect combustion tube	SA
RADIOCHEMISTRY	
LOW BACKGROUND GAS PROPORTIONAL COUNTERS	
Clean sample drawers and windows	W
LUCAS CELLS	
Clean Windows	M
REFRIGERATORS, INCUBATORS, OVENS	
Clean interior	M
Check thermometer temperature against certified thermometer or equivalent	A
ANALYTICAL BALANCES	
Clean pan and compartment	D
Check with class S weights	M
Manufacturer cleaning and calibration	A
AUTOCLAVES	
Gaskets checked	W(1)
Timing mechanism checked	SA
Clean interior	M
Sterilization indicator tape	D

TABLE 8.1, continued
PREVENTIVE MAINTENANCE ACTIVITIES

INSTRUMENT/ACTIVITY	FREQUENCY
MICROSCOPES	
Clean optics	M
UV/VIS SPECTROPHOTOMETER	
Lamp alignment checked	3,4
Windows cleaned	M
Check and adjust photomultiplier sensitivity and wavelength resolution	A
Replace lamp	4
Clean sample compartment before and after each use	D
Check electronics	A(3,4)
Adjust baseline for smoothness through entire wavelength range	SA
Clean cuvettes after each use	D
TOX ANALYZER (TOTAL ORGANIC HALOGENS)	
Clean titration cell; clean inlet and exit tube	W
Clean pyrolysis tube, recoat electrodes	5
pH AND ION SELECTIVE ELECTRODES	
PROBE	
Check probe for cracks and proper levels of filling solution; check reference junction; clean electrode	D(1)
Check response time	D
METER	
Check batteries and electronics for loose connections and cracked leads	D(1)
TURBIDIMETER	
Clean instrument housing	M
Clean cells	D
CONDUCTIVITY METER	
Check batteries and probe cables	D
Replatinize Probe	5
DISSOLVED OXYGEN METERS	
PROBE	
Check membrane for deterioration; check filling solution	D(1)
METER	
Battery level and electronics checked	D(1)

TABLE 8.1, continued
 PREVENTIVE MAINTENANCE ACTIVITIES

INSTRUMENT/ACTIVITY	FREQUENCY
THERMOMETERS	
Check for cracks and gaps in the mercury	D(1)
TEMPERATURE PROBE	
Check connections, cables	D
Check against calibrated thermometer	D
AUTOSAMPLERS	
Check needles and tubing	D(1)
Clean	Q
DATA SYSTEMS	
Clean computers, check battery backup and check ventilation fans	Q
AUTOMATIC SAMPLE COLLECTION SYSTEMS (ex. Isco, Sigma, etc.)	
Check sampler operation (forward, reverse, automatic through three cycles of the purge-pump-purge cycle)	D(6)
Check purge-pump-purge cycle when sampler is installed	D(7)
Check the flow pacer that activates the sampler to assure proper operation	D(6)
Check desiccant	D(1,6)
Check batteries	D(1,6)
Check pumping rate against manufacturer's specifications	D(1,6)

KEY:

1 Replace as necessary	D daily*
2 High background	W weekly
3 Loss of sensitivity or failing resolution	M monthly
4 Erratic response	Q quarterly
5 QC failure	SA semi-annually
6 Prior to sampling event	A annually
7 In situ (under field conditions)	

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

**9.0 MINIMUM QUALITY CONTROL REQUIREMENTS AND ROUTINES TO
CALCULATE AND ASSESS PRECISION, ACCURACY AND METHOD DETECTION
LIMITS**

9.1 QC CHECKS

9.1.1 Minimum Field Quality Control Requirements

Field Quality Control activities shall meet or exceed the following requirements.

9.1.1.1 Quality Control Checks

a. Field Quality Control Blanks

1. General Considerations:

- a. All equipment blanks shall be collected and analyzed for the same parameters as the associated samples.
- b. All blanks shall be preserved, transported, documented and handled as if they were samples. Once collected, they must remain with the sample set until they have been received by the laboratory.
- c. All equipment blanks are prepared by rinsing the sampling equipment with analyte-free water and collecting the rinsate in appropriate sample containers (see a and b above).

The following types of blanks shall be collected as specified:

- a. **Precleaned Equipment Blank:** These blanks shall be collected from sampling equipment that has been brought to the site precleaned and ready for use. At least one equipment blank shall be collected for each water and solid matrix analytical group. These blanks shall be collected **AT THE BEGINNING** of the sampling episode.
- b. **Field Cleaned Equipment Blank:** These blanks shall be collected from sampling equipment **AFTER** the equipment has been cleaned in the field (i.e., between sampling points).
- c. **Trip Blank:** These blanks are required only if samples are to be analyzed for VOCs. They shall be prepared by the organization that is providing the VOC vials, and shall be prepared by filling vials with analyte-free water. The vials shall be placed in the same transport containers as the empty VOC vials. They must remain with the VOC vials during the sampling episode and shall be transported to the

analyzing laboratory in the same shipping or transport container(s) as the VOC samples. The trip blanks shall remain **UNOPENED** for the entire sampling episode. A trip blank must be submitted for each cooler that transports empty or full VOC vials.

b. Field Duplicates

1. Field duplicates shall be collected and analyzed for the same parameters as the associated samples.

2. They shall be preserved, transported and documented in the same manner as the samples. **THESE SAMPLES ARE NOT CONSIDERED LABORATORY DUPLICATES.**

3. Duplicates are collected to measure the variability inherent in the sampling process. They shall be obtained by DUPLICATING (simultaneously or in rapid succession) the entire sample acquisition technique that was used to obtain the first sample.

a. Duplicates for water are collected by sampling from successively collected volumes (i.e., samples from the next bailer of sample water).

b. Duplicates for soils are collected from the same sample source (i.e., soil is obtained from the same soil sampling device).

c. Frequency

1. The frequency with which the above quality control samples are collected is summarized below:

# of Samples	Precleaned Equipment BLK minimum of one then 5%	Field cleaned Equipment BLK minimum of one then 5%	Trip BLK (VOCs) one per cooler	Duplicate
				minimum one then 10%
10+				
5-9	one*	one*	NR	one
< 5	one*	one*	NR	NR

NR=Not Required

BLK=Blank

* Note: For 9 or fewer samples, a precleaned equipment blank or a field cleaned equipment blank is required. A field cleaned equipment blank must be collected if equipment is cleaned in the field.

2. Clarification and Explanation

a. The number and type of equipment blanks (EQB) are dependent upon two factors above and beyond the number of samples taken at a given site.

1. The first factor is the number of precleaned pieces of sampling equipment (bailer, Nansen bottle, Kemmerer, etc.) brought into the field for use.
2. The second factor is the total number of field cleanings performed on this equipment.
 - b. To calculate the number of each kind of EQB (precleaned and field cleaned) the following sequence should be used:
 1. Determine the total number of samples taken for each matrix.
 2. Determine the total number of pieces of equipment by type to be used for a given matrix which will be brought into the field precleaned.
 3. Determine the total number of field decontaminations that will be necessary for the sampling event by subtracting #2 from #1 above.
 4. Once these have been determined, the requirements on the following table and the generalities listed below may be used to determine the total number of EQBs that must be collected and analyzed:
 - a. For 1 to 9 samples, at least one EQB must be taken, either Precleaned OR Field cleaned. If field cleaning is performed, then the EQB must be a Field cleaned EQB.
 - b. For 10 to 20 samples, one Precleaned EQB AND one Field cleaned EQB must be taken unless all equipment used is precleaned. If no field cleaning is performed, then only one Precleaned EQB is required.
 - c. For greater than 20 samples, the 5% (1 in 20) requirement must be met for BOTH the Precleaned (number 2 above) and Field cleaned (number 3 above) equipment.

Examples for sample sets greater than 20:

<u>Total Samples Taken</u>	<u>Total Pre-Clean Equip. Used</u>	<u>Required Field Cleanings</u>	<u>Precleaned EQB # Required</u>	<u>Field Cleaned EQB# Required</u>
25	1	24	1	2
	5	20	1	1
	21	4	2	1
	25	0	2	0
30	1	29	1	2
	10	20	1	1
	21	9	2	1
	30	0	2	0

<u>Total Samples Taken</u>	<u>Total Pre-Clean Equip. Used</u>	<u>Required Field Cleanings</u>	<u>Precleaned EQB # Required</u>	<u>Field Cleaned EQB# Required</u>
35	1	34	1	2
	15	20	1	1
	21	14	2	1
	35	0	2	0

Note: To reduce the number of submitted EQBs, bring sufficient precleaned equipment into the field such that 20 or fewer field cleanings are performed.

9.1.1.2 Split Samples

a. DER or the client may require split samples as a means of determining compliance or as an added measure of quality control. These type of samples are intended to measure the variability between laboratories and should be obtained as subsamples from the same parent sample.

b. A true split sample of soil, sediment or sludge is almost impossible to accomplish under field conditions.

c. Split samples shall be collected, preserved, transported and documented using the same protocols as the related samples. In addition, an attempt should be made to use the same preservatives (if required).

d. Split samples for water shall be collected in one of two ways:

1. Mix the sample in a large, appropriately precleaned, intermediate vessel and pour aliquots of the mixed sample into the appropriate sample containers. This method shall not be used if VOCs are of interest.

2. Fill the sample containers from consecutive sample volumes FROM THE SAME SAMPLING DEVICE (i.e., from the same bailer). If the sampling device does not hold enough sample to fill the sample containers, the following protocol shall be used:

- a. Fill the first container with half of the sample, and pour the remaining sample into the second container.

- b. Obtain additional sample, and pour the first half into the SECOND container. The remaining portion shall be poured into to first container.

- c. Continue with steps 1 and 2 until both containers are filled.

9.1.1.3 Quality Control on Field Measurements

a. All field instruments must be initially calibrated at the beginning of each working day.

- b. A continuing calibration check shall be analyzed at intervals of no more than 4 hours and at the end of the sampling day to determine if the instrument has maintained calibration.
- c. The instrument shall be recalibrated if the continuing calibration checks fail to meet acceptance criteria.
- d. All quality control data shall be recorded in the daily field notes.

9.1.2 Laboratory QC Checks

The laboratory shall follow the minimum quality control requirements specified by each method. If no quality control requirements are listed in the method, or if the method quality control requirements are less stringent than those listed below, the laboratory shall follow the guidelines listed below:

9.1.2.1 Chemistry QC Checks

- a. Method reagent blanks - shall be prepared and analyzed at a rate of one per sample set (see definitions in Appendix A).
- b. Matrix Spikes - At least one sample in a sample set (or 5%, whichever is greater) with similar matrices shall be prepared and analyzed by the specified method. If a set contains samples of different matrices, matrix spikes should be prepared and analyzed for each matrix type. For work submitted to DER, matrix spikes must be included as routine protocol.
- c. Reagent water or reagent matrix spikes - Reagent water or reagent matrix spikes may be used as additional QC checks to monitor the effectiveness of the method. If used, these must be analyzed at a frequency of 5%.
- d. Quality control check samples - shall be analyzed in duplicate semiannually. Such samples shall be analyzed as blind samples (i.e., the component concentrations in these samples shall not be provided to the analyst until after analysis). If the data are not acceptable, the analytical results must be reported in a QA report to DER (see Section 13).
- e. Quality control check standards - shall be analyzed at a continuing frequency equivalent to 5% of the samples in the analytical set (i.e. one every 20 samples) or shall be analyzed at the beginning of each run to verify the standard curve.
- f. Duplicate samples or matrix spike duplicates - at least one or 5% of all samples in a sample set with a similar matrix shall be selected and analyzed in duplicate. If a sample set contains samples from different matrices (e.g.,

effluent and drinking water), then duplicates or matrix spike duplicates should be analyzed for each matrix.

g. Continuing calibration standards shall be analyzed at a frequency equivalent to 5% of the samples in an analytical set. Alternatively, quality control check standards may be used (see e. above). At least one of these checks shall be a standard at a concentration of 1 - 2 times the laboratory stated PQL.

h. Additional quality control checks may be included and shall be used if specified by the approved method:

- a. Reagent purity checks
- b. Internal standards
- c. Surrogate spikes

9.1.2.2 Microbiology QC Checks

a. Blanks

1. Membrane Filter Analysis: For each set of samples, a control blank shall be run at the beginning (dilution water blank), every tenth sample (sample carry over blank), and at the end of the set.

2. MPN Analysis: A single tube of LTB broth media shall be inoculated with 10 milliliters of sterile phosphate buffered dilution water (dilution blank control).

b. Duplicates - At least 10% of the known positive samples that have been processed shall be analyzed in duplicate or a minimum of one duplicate analysis per month for MF and MPN analyses.

c. Positive/Negative Controls - Microorganisms obtained from the American Type Culture Collection (ATCC) or equivalent sources shall be used to confirm the morphological and biochemical responses to test media. Positive and negative controls shall be run with each new lot of media, and monthly thereafter.

d. Water Quality Indicators:

1. Water source shall be tested monthly for chlorine residual, conductivity and standard plate count.

2. The concentration of metals in the water source shall be determined annually.

3. Water Suitability Test shall be conducted annually.

e. 5% of all positive environmental samples analyzed by membrane filter shall be verified per method requirements.

f. When using the MPN test, the MPN test shall be completed on 10% of all positive confirmed samples.

9.1.2.3 Laboratory QC Checks (Bioassays)

a. At least one set of controls (dilution water and hardness or salinity, if appropriate) shall be run with each test.

b. Analytical equipment shall follow the chemistry laboratory quality control checks listed above.

9.1.2.4 Laboratory QC Checks (Species Identification)

- a. Should maintain or have access to a type specimen collection.
- b. Must, at a specified frequency use outside experts to corroborate species identification.

9.2 ROUTINE METHODS USED TO ASSESS PRECISION AND ACCURACY

9.2.1 Precision and accuracy targets listed in the tables of Methods, Matrices and QA Targets (Section 5.0 of a CompQAP) must be generated from matrix spikes and matrix spike duplicates or duplicates of environmental samples. The laboratory must maintain a list of QC checks, as presented in Section 9.1.2, which identifies applicable analytical methods and the concentrations to be used to make the determination in terms of low, mid or high levels:

1. Low level is defined as concentrations from the minimum detection limit to a level 5 times the MDL.
2. Mid level is defined as the mean level between the minimum detection level and the upper end of the linear range.
3. High level is defined as concentrations at the upper end of the linear range.

9.2.3 The laboratories shall use the following formulas for calculating the precision and accuracy of test measurements and the associated acceptance ranges:

9.2.3.1 The precision of replicate pairs shall be calculated using one of the following formula:

- a. The precision of duplicate pairs shall be calculated using
 1. Percent Relative Standard Deviation (% RSD)

$$\% \text{ RSD} = \frac{s}{\bar{x}} \times 100$$

Where: \bar{x} = Mean (average) of the data points
 s = Standard deviation calculated as:

$$\left[\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1} \right]^{1/2}$$

In the case of pairs (duplicates) this formula becomes:

$$\% \text{ RSD} = \frac{|A-B|}{A+B} \times \frac{2}{\sqrt{2}} \times 100$$

2. Relative Percent Difference (RPD)

$$RPD = \frac{|A-B|}{A+B} \times 200$$

Where: A = concentration in sample A
 B = concentration in sample B

3. Industrial Statistic (I)

$$I = \frac{|A-B|}{A+B}$$

Where: A = concentration in sample A
 B = concentration in sample B

9.2.3.2 The accuracy of a measurement shall be determined by the recovery of a known amount of analyte in a real sample as:

$$\% R = \frac{C_s - C_u}{S} (100)$$

Where: C_s = concentration of spiked sample
 C_u = concentration in unspiked sample
 S = expected concentration of spike in sample
 $\%R$ = percent recovery

9.2.3.3 The accuracy of a measurement based on known concentrations (i.e. performance evaluation samples) shall be calculated as:

$$\% R = \frac{\text{Sample Concentration} (100)}{\text{Reported True Value}}$$

9.2.3.4 Upper and Lower Warning and Control Limits to be used as acceptance criteria shall be calculated as follows:

$$S = \left[\frac{\sum_{i=1}^n (x - \bar{x})^2}{n-1} \right]^{1/2}$$

Where: S = Standard Deviation
 n = Number of points or data pairs to be included in the calculation
 x = Sample Percent Recovery or precision of replicates
 \bar{x} = Mean (average) of the data points

$$CL = P_{av} \pm 3 S$$

Where: CL = Control limit (upper and/or lower)
 P_{av} = Mean of P (average percent recovery or average % RSD)

$$WL = P_{av} \pm 2 S$$

Where: WL = Warning limit (upper and/or lower)

9.2.3.5 Microbiological quality control acceptance criteria shall be calculated per formulae specified in Standard Methods for the Examination of Water and Wastewater, 17th Edition, Method Number 9020, Section 4.b (pp. 9-17 and 9-18).

9.2.4 Quality Control charts must be prepared or other easily followed system instituted to track results of QC checks.

9.2.5 Quality control charts or tabulation systems must be updated every 20 data points or annually at a minimum.

9.3 METHOD DETECTION LIMITS AND PRACTICAL QUANTITATION LIMITS

9.3.1 Method Detection Limits (MDLs)

MDLs shall be determined by one of three protocols:

1. EPA - "Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11", 40 CFR Part 136, Appendix B;
2. IUPAC - "Nomenclature, symbols, units and their usage in Spectrochemical analysis II," Spectrochim Acta B 1978, 33B, 242 and "Limit of Detection, A Closer Look at the IUPAC Definition," Analytical Chemistry 1983, V. 55, No. 7 page 712A;
3. USATHAMA - "Decision and detection limits for linear calibration curves," Hubaux, A. and Vox, G., Analytical Chemistry 42, 849 - 855 (1970).

IN ALL CASES, THE METHOD DETECTION LIMIT IS DEFINED TO BE THREE TIMES THE STANDARD DEVIATION DERIVED FROM THE STUDY.

9.3.2 Practical Quantitation Limit (PQLs)

The PQL is defined as 12 times the standard deviation that is derived from the procedures used to determine MDL.

Note: EPA is currently considering changing the names BUT NOT THE DEFINITIONS of the above-mentioned terms:

MDL is proposed to be Method Detection Level (currently method detection limit); and

RDL is proposed to be Reliable Quantitation Level (currently practical quantitation limit).

9.3.3 MDLs or PQLs shall be verified on an continuous basis (see 9.1.2.1.g).

9.4 DOCUMENTATION

9.4.1 See the Custody Section for Field QC checks.

9.4.2 Laboratory Checks

1. Records which document sample/standard preparation, source and concentration (this includes protocols for preparation and certification, if applicable) must be maintained. All required records specified in the Calibration SOP must be maintained.
2. Identification of analyses set that the applicable QC sample is associated with.
3. Calculations performed to determine QC results.
4. Control limits used to evaluate analysis results and how these were determined.

9.4.3 MDL Studies

1. Documentation for the MDL studies must be conducted according to all other SOPs regarding sample and standard handling procedures, calibrations, QC checks and analyses.
2. Documentation must include:
 - a. Date of studies and calendar period for which the studies are applicable
 - b. Analytical Method
 - c. Identification of analyst responsible for analysis
 - d. Compounds studied

10.0 DATA REDUCTION, VALIDATION AND REPORTING

10.1 DATA REDUCTION

Data reduction includes all activities that convert instrument/computer responses into reportable results. These activities may involve mathematical calculations, compound identification and summary statistics. The final results may be obtained in two ways:

1. Direct readings from the instrument; or
2. Calculations based on instrument output, readings or responses.

The initial data reduction is the responsibility of the analyst or field technician who operates the analytical instrument. In addition to the general duties specified below, additional responsibilities for manual and computer related data reduction have been specified.

1. Calculate spike recoveries and precision for duplicates;
2. Identify quality control data (blank, spikes, duplicates, etc.) for review by quality assurance officer;
3. Assure accurate transcription of sample identification numbers on all records;

10.1.1 Manual Data Reduction

1. If applicable, assure that all readings or output are precisely measured and noted on strip charts;
2. Select appropriate formulae for calculating final results;
3. Enter the formulae and at least one complete sample calculation on the strip chart or in the notebook;
4. Assure that all data are accurately transcribed into notebooks, forms or spreadsheets;
5. Enter all manual calculations into notebook or data records;
6. Check raw data entries with final computer output to assure accurate initial data entry;
7. Record appropriate and accurate information concerning sample identification, operating conditions, etc.

If raw data is entered into a computer program or spreadsheet for data reduction, the organization must be aware of and have on file a record of the mathematical formulae that are being used by the computer. If such information is not available, the organization shall verify the formula by manual calculations and maintain a record of the verification process.

All raw data output (strip charts, tabular printouts, etc.) must be retained as a part of the records. These records at a minimum must be identified with the following information: Date of run; sample ID numbers; analyst or operator; type of analysis

(nitrate, metals, etc.). In addition, the following information must be maintained: instrument operating conditions (if applicable); detector and column types; instrument configuration; etc. The latter information may be kept in cross referenced records or may be entered on the various output records.

10.1.2 Computer/Integrator Reduction

1. Assure that all data to be used in final calculations are entered accurately: sample weights or volumes; final extract volumes; dry weight factors; dilution factors; surrogate standard concentrations, etc.;
2. Properly interpret the computer output in terms of properly identified components, positive or negative identifications, and appropriate confirmatory measures;
3. Record appropriate and accurate information concerning sample identification, operating conditions, etc.;
4. Calculate surrogate recoveries and internal standard responses (if applicable);
5. GC and GC/MS analyses should be checked to verify that target components are within acceptable retention time windows and that additional confirmation (if needed) is initiated.

Many analytical instruments are interfaced with computers or integrators that automatically evaluate, identify and calculate final values. The results are printed in combinations of graphic (ex. chromatograms) and tabular forms. As with manual data reduction, the organization must be aware and should on file a record of the mathematical formulae or algorithms that are being used by the computer. If the information is not available, the organization shall maintain records which demonstrate that the software is providing the expected results (e.g. check sample or check standard data is acceptable).

Typically computer data files are identified by a queue number or a data file number. In such cases, the organization must maintain a cross reference index or log to identify the computer data files with sample ID numbers. Additional information that should be entered into the data file records are: date of run, analysis type, and analyst initials. Cross referenced auxiliary records are required to identify instrument operating conditions (if applicable); detector and column types; instrument configuration; etc.

10.1.3 Formulae and Calculations

The final results of each test shall be calculated by the formula specified in the analytical method that is being used.

The final result should be rounded off to an appropriate number of significant figures (typically 2 significant figures).

If the digit 6,7,8 or 9 is dropped, increase the preceding digit by one unit; if the digit 0,1,2,3, or 4 is dropped, do not alter the preceding digit. If the digit to be dropped is 5, round off the preceding digit to the nearest even number: 2.25 becomes 2.2 and 2.35 becomes 2.4.

As a general rule the results should be converted to the reporting units presented on Table 10.1. Other reporting conventions (i.e. wet weight instead of dry weight) should be clearly identified on the final reports with appropriate justification.

Note: If components of interest are detected in any quality control blank(e.g. method blanks, digestion blanks, etc.), the blank concentration must be reported. The blank concentration shall not be subtracted from any associated sample data.

10.2 DATA VALIDATION

10.2.1 Data Integrity

Data integrity involves checking all field and laboratory data entries and calculations for errors and mistakes. It also involves reviewing all documentation to assure that sample ID numbers are correct, and that the tests have been performed within appropriate holding times. Data integrity does not include assessment of quality control measures.

All data integrity checks should be performed by an individual (preferably a supervisor) who was not involved in the original data reduction process.

The following records and documentation must be checked:

1. A minimum 10 - 20% of raw data entries for transcription accuracy;
2. A minimum 10 - 20% of all calculations randomly checked for mathematical errors including dilution factors, final volumes, dry weight factors and sample volumes or weights. Note: field calculations for well and purge volume are excluded;
3. Verify acceptability of initial and/or continuing calibration data;
4. Sample preparation logs and instrument or analytical logs to assure that samples were prepared and analyzed within prescribed holding times;
5. Cross check all records for completeness and for transcription errors associated with the sample number;
6. Verify all chain of custody records (if applicable) for completeness and acceptability;

TABLE 10.1
DATA REPORTING UNITS

TEST NAME OR COMPONENTS	REPORTING UNITS			
	<u>WATER</u>	<u>SED</u>	<u>FISH</u>	<u>WASTE</u>
Metals <u>except</u> :	ug/L	mg/kg	mg/kg	mg/kg
Reports for potable (drinking water), Calcium, magnesium, sodium, potassium	mg/L	mg/kg	mg/kg	mg/kg
Purgeable organic components (VOCs and VOAs)	ug/L	ug/kg	mg/kg	mg/kg
Extractable organic components Including pesticides and herbicides <u>except</u> :	ug/L	ug/kg	mg/kg	mg/kg
Dioxin/Furan Scan and Dibenzo dioxins and dibenzofurans	ng/L	ng/kg	ng/kg	ng/kg
Odor (60 Degree C)	TON			
Odor (Room Temp)	TON			
pH (Laboratory)	pHUN			
Color (True-PTCO)	CU			
Color (Apparent-PTCO)	CU			
Conductivity	uMHOS/cm			
Corrosivity				
Flash Point				DEG F
Hardness (as CaCO3)	mg/L			
Settleable Solids	ml/L/h			
Total Solids	mg/L	mg/kg		mg/kg
Volatile Total Solids	mg/L	mg/kg		mg/kg
Total Suspended Solids	mg/L	mg/kg		mg/kg
Volatile Total Suspended Solids	mg/L	mg/kg		mg/kg
Total Dissolved Solids (180 Degree C)	mg/L			
Volatile Total Dissolved Solids	mg/L	mg/kg		mg/kg
Toxicity (EP and TCLP)	mg/L			
Turbidity	NTU			
Radium-226, Total	pc/L	pc/g		
Radium-228, Total	pc/L	pc/g		
Radium-226, Diss	pc/L	pc/g		
Radium-228, Diss	pc/L	pc/g		
Gross Alpha, Total	pc/L	pc/g		
Gross Beta, Total	pc/L	pc/g		
Acidity	mg/L	mg/kg	mg/kg	mg/kg

TABLE 10.1
DATA REPORTING UNITS (continued)

TEST NAME OR COMPONENTS	REPORTING UNITS			
	WATER	SED	FISH	WASTE
Alkalinity, Bicarbonate (as CaCO ₃)	mg/L			
Alkalinity, Carbonate (as CaCO ₃)	mg/L			
Alkalinity, Total (as CaCO ₃)	mg/L	mg/kg	mg/kg	mg/kg
Bicarbonate (as HCO ₃ ION)	mg/L	mg/kg		
Carbonate (as CO ₃ ION)	mg/L	mg/kg		
Ammonia (as N)	mg/L	mg/kg	mg/kg	mg/kg
Ammonia, Dissolved (as N)	mg/L			
Ammonia, Unionized (as NH ₃)	mg/L			
Bromide	mg/L			
Chloride	mg/L	mg/kg		mg/kg
Chlorine Residual	mg/L	mg/kg	mg/kg	mg/kg
Cyanide	mg/L	mg/kg	mg/kg	mg/kg
Cyanide Amenable to Chlorination	mg/L	mg/kg	mg/kg	mg/kg
Cyanide, Free	mg/L	ug/kg	mg/kg	mg/kg
Dissolved Oxygen (Winkler)	mg/L			
Dissolved Oxygen (Electrode)	mg/L			
Fluoride	mg/L	mg/kg	mg/kg	mg/kg
Nitrate-Nitrogen	mg/L	mg/kg	mg/kg	mg/kg
Nitrite-Nitrogen	mg/L	mg/kg		
Nitrate+Nitrite Nitrogen	mg/L	mg/kg	mg/kg	mg/kg
Nitrate+Nitrite Nitrogen, Dissolved	mg/L			
Ortho-Phosphate Phosphorus	mg/L	mg/kg	mg/kg	mg/kg
Silicon (Si)	mg/L	mg/kg	mg/kg	mg/kg
Silica (SiO ₂)	mg/L	mg/kg	mg/kg	mg/kg
Sulfate	mg/L	mg/kg		mg/kg
Reactive Sulfides (as H ₂ S)	mg/L	mg/kg		mg/kg
Sulfides	mg/L	mg/kg		mg/kg
Sulfite	mg/L			
Temperature	Deg C			
Total Dissolved Phosphorus	mg/L			
Total Kjeldahl Nitrogen	mg/L	mg/kg	mg/kg	mg/kg
Total Kjeldahl Nitrogen, Dissolved	mg/L			
Total Phosphorus	mg/L	mg/kg	mg/kg	mg/kg
Bio-Chemical Oxygen Demand, 5 Day	mg/L	mg/kg		mg/kg

TABLE 10.1
DATA REPORTING UNITS (continued)

TEST NAME OR COMPONENTS	REPORTING UNITS			
	WATER	SED	FISH	WASTE
Bio-Chemical Oxygen Demand, 5 Day, Dissolved	mg/L	mg/kg		mg/kg
Bio-Chemical Oxygen Demand, 20 Day	mg/L	mg/kg		mg/kg
Bio-Chemical Oxygen Demand, 60 Day	mg/L	mg/kg		mg/kg
BOD, Carbonaceous, 5 Day	mg/L			
Chemical Oxygen Demand	mg/L	mg/kg		mg/kg
Chemical Oxygen Demand, Dissolved	mg/L			
Linear Alkyl Sulfonate	mg/L	mg/kg	mg/kg	mg/kg
Oil and Grease	mg/L	mg/kg	mg/kg	mg/kg
Phenols (4AAP)	ug/L	mg/kg	mg/kg	mg/kg
Total Organic Carbon	mg/L	mg/kg	mg/kg	mg/kg
Total Organic Carbon, Dissolved	mg/L			
Purgeable Organic Carbon	mg/L			
Total Organic Halogen	ug/L	ug/kg		mg/kg

10.2.2 Data Validation

Data validation is accomplished through a series of checks and reviews that are intended to assure that the reported results are of a verifiable and acceptable quality.

A majority of these tasks should be performed by the Quality Assurance Officer, but some may be delegated to another individual (ex. supervisor) who was not actively involved with generating the data.

1. Verify that all quality control blanks meet criteria;
2. Review all other quality control data (spikes, duplicates, quality control check standards, quality control check samples, etc.) for acceptability;
3. Review all surrogate and standard additions spike recoveries and internal standard responses for acceptability;
4. Identify any sample set or data that are unacceptable and initiate appropriate corrective action measures;
5. Assign data qualifiers (if needed) to reported values;
6. Verify mass spectral interpretation (if applicable) and/or component identification;
7. Assign data qualifiers to all applicable data (see Table 10.2). Note: the reported value always precedes the data qualifier code.

10.3 DATA REPORTING AND OVERALL PROJECT VALIDATION

10.3.1 Laboratory Data Reports

The final reports from the laboratory may be generated in several different ways:

1. Hand written report forms;
2. Manually typed reports and narrative;
3. Computer generated reports;
4. Any combination of the above methods.

All parties who are involved with the data review and validation process are responsible for providing data entry operators or clerical personnel with accurate records for transcription. If data are automatically reported through a LIMS system, the final reviewer must assure that the appropriate commands have been input to release the data for final reports.

10.3.2 Engineering Project Reports

The final reports from an engineering firm or the organization responsible for a project involves assimilating and presenting data from both the laboratory and field. These reports may also

TABLE 10.2
DATA QUALIFIER CODES

SYMBOL	MEANING
A	Value reported is the mean (average) of two or more determinations. This code shall be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed (e.g. laboratory replicate samples, field duplicates, etc.) independently. Do not use this code if the data are the result of replicate analyses on the same sample aliquot, extract or digestate. Under most conditions, replicate values shall be reported as individual analyses.
B	Results based upon colony counts outside the acceptable range. This code applies to microbiological tests and specifically to membrane filter colony counts. The code is to be used if the colony count is generated from a plate in which the total number of coliform colonies EXCEEDS the method indicated ideal ranges which are: Total Coliforms: 20 - 80 colonies Fecal Coliforms: 20 - 60 colonies
F	When reporting species: F indicates female sex.
H	Value based on field kit determination; results may not be accurate. This code shall be used if a field screening test (i.e. field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods.
J	Estimated value; value not accurate. This code shall be used in the following instances: 1. surrogate recovery limits have been exceeded; 2. no known quality control criteria exists for the component; 3. the reported value failed to meet the established quality control criteria for either precision or accuracy; 4. the sample matrix interfered with the ability to make any accurate determination; or 5. the data is questionable because of improper laboratory or field protocols (e.g. composite sample was collected instead of a grab sample). Note: 1. A "J" value shall be accompanied by justification for its use. 2. A "J" value shall not be used if another code applies (ex. K, L, M, T, V, Y, PQL)

TABLE 10.2
 DATA QUALIFIER CODES (continued)

SYMBOL	MEANING
	<p>Note: 1. A "J" value shall be accompanied by justification for its use.</p> <p>2. A "J" value shall not be used if another code applies (ex. K, L, M, T, V, Y, PQL)</p>
K	<p>Off-scale low. Actual value is known to be less than the value given. This code shall be used if:</p> <ol style="list-style-type: none"> 1. The value is less than the lowest calibration standard AND the calibration curve is known to be non-linear; or 2. The value is known to be less than the reported value based on sample size, dilution or some other variable. <p>This code SHALL NOT be used to report values that are less than the laboratory practical quantitation limit or laboratory method detection limit.</p>
L	<p>Off-scale high. Actual value is known to be greater than value given. To be used when the concentration of the analyte is above the acceptable level for quantitation (exceeds the linear range or highest calibration standard) <u>AND</u> the calibration curve is known to exhibit a negative deflection.</p>
M	<p>When reporting chemical analyses: presence of material is verified but not quantified. The reported value shall be the laboratory practical quantitation limit. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is GREATER THAN the laboratory method detection limit. If the value is less than the method detection limit, use "T", below.</p> <p>When reporting Oxygen Reduction Potential or Temperature: indicates a negative value</p> <p>When reporting Species: indicates male sex.</p>
N	<p>Presumptive evidence of presence of material. This qualifier shall be if:</p> <ol style="list-style-type: none"> 1. the component has been tentatively identified based on mass spectral library search; 2. if the presence of the analyte is indicated but there is evidence of possible interferences; or 3. there is an indication that the analyte is present, but quality control requirements for

TABLE 10.2
DATA QUALIFIER CODES (continued)

SYMBOL	MEANING
	confirmation were not met (i.e. presence of analyte was not confirmed by alternate procedures).
O	Sampled, but analysis lost or not performed. Note: if reporting data to STORET, a numerical value must be entered. Such values are not meaningful and shall not be used.
Q	Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared and/or analyzed AFTER the approved holding time restrictions for sample preparation and analysis.
T	Value reported is less than the laboratory method detection limit. The value is reported for informational purposes only and SHALL NOT be used in statistical analysis.
U	Indicates that the compound was analyzed for but not detected. This shall be used to indicate that the specified component WAS NOT detected. The value associated with the qualifier shall be the laboratory method detection limit. Unless requested by the client, less than the method detection limit values shall not be reported (see "T" above).
V	Indicates that the analyte was detected in both the sample and the associated method blank. Note: the value in the blank shall not be subtracted from associated samples.
Y	The laboratory analysis was from an unpreserved or improperly preserved sample. The data may not be accurate.
Z	Too many colonies were present (TNTC), the numeric value represents the filtration volume.
PQL	The reported value is between the laboratory method detection limit and the laboratory practical quantitation limit.

TABLE 10.2
 DATA QUALIFIER CODES (continued)

SYMBOL	MEANING
REJ	Data is rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.
NAI	Not analyzed due to interference.

If more than one code applies, and the data is to be entered into STORET, only one code shall be reported. The code shall be selected based on the following hierarchy:

- REJ
- NAI, O
- Y
- V
- H
- J
- B, K, L, M, PQL, T, Z
- A

The following codes deal with certain aspects of field activities. The codes shall be used IF the laboratory has knowledge of the specific sampling event. The codes shall be added by the organization collecting the sample, if they apply:

D	Measurement was made in the field (i.e. in situ). This applies to any value (ex pH, specific conductance, etc.) that was obtained under field conditions using approved analytical methods. Note: when data is to be entered into STORET, and the parameter code specifies a field measurement (e.g. "Field pH"), this code is not required.
E	Indicates that extra samples were taken at composite stations.
R	Significant rain in the past 48 hours. This code shall be used when the rainfall might contribute to a lower than normal value.
HIS	Data deviates from historically established concentration ranges.

include narratives on site history, an analysis of current findings; and conclusions and/or recommendations on further project work.

10.3.3 Project Validation

Project validation is the process by which all project data is reviewed prior to reporting the data to the client. This task is normally assigned to the project manager but may be performed by an individual who is responsible for overall management operations.

10.3.3.1 Laboratory

- a. Review all identified quality control checks. Assure that any deviations or questionable data have been reported with qualifiers or with appropriate explanations;
- b. Check for overall project consistency and any obvious anomalous values;
- c. Check for clerical errors, transposed numbers and accurate data transfer.

10.3.3.2 Field/Engineering

- a. Review all quality control data (field and laboratory) for project acceptability. Attach appropriate justification or explanation for any questionable data;
- b. Check for overall project consistency, including comparison with historical or expected results;
- c. Check for clerical errors, transposed numbers and accurate data reporting.

All final reports should be verified and signed by the project manager(s), laboratory director or other individual who is responsible for the overall operations of the organization.

10.4 DATA STORAGE

All records of an organization that are pertinent to a specified project must be retained for a period of at least 3 years after the completion of the project. These records include:

1. All field notebooks, data sheets and documentation on the sampling event;
2. All field and laboratory analytical records including supporting calibration, raw data, data reduction calculations, quality control information and all data output records (chromatograms, strip charts and other instrument response readout records);
3. All field and laboratory custody records including shipping receipts, sample transmittal forms, internal routing and assignment records and sample disposal;
4. All notebooks, data forms, and logs pertaining to laboratory operations including sample receipt and log in;

5. All records concerning receipt, preparation and use of calibration standards;
6. All statistical calculations used in data reduction and in determination of quality control limits;
7. Preventative maintenance records for all analytical and support equipment and instrumentation;
8. Copies of final reports. Note: reports for drinking water laboratories must comply with Chapter 17-550 which requires retention times of up to 10 years.

Records that are stored by computers or PCs must have hard copy and write-protected backup copies.

The records must be protected from environmental degradation; stored under secure conditions to discourage tampering or vandalism; and must be cross indexed by project number, laboratory ID number or some other common identifier for easy retrieval.

11.0 CORRECTIVE ACTIONS

Quality controls are used to monitor and assess the effectiveness and validity of a sampling or analysis activity. If a specified quality control measure is determined to be out of a predetermined acceptance range, and the source or reason for the deviation is not identified and corrected, the sample data associated with the quality control measure may not be useful or valid information.

Some quality control criteria (ex. calibration) have a direct effect on the test results. Others (ex. blanks and duplicates) are indicators of improper protocols or contamination.

11.1 QUALITY CONTROL MEASURES AND ACCEPTANCE CRITERIA

Table 11.1 identifies each of the quality control checks that are required by test methods and/or DER acceptance criteria. The acceptance range criteria or the source of the acceptance range has been identified.

11.2 IDENTIFYING AND ASSESSING QC MEASURES

Generally, quality control information is reviewed by several individuals. The responsibility for the initial assessment of a quality control measure lies with the individual who (1) identifies the sample or procedure as a QC measure; and (2) has access to the test results:

11.2.1. The individual responsible for operating the analytical instrument or equipment must be responsible for assessing the following applicable QC Measures:

1. Method, reagent and calibration blanks
2. Calibration integrity: initial and continuing calibration, interference standards, and QC check standards
3. System performance checks
4. Tuning criteria
5. Surrogate and internal samples
6. Titrating solutions

11.2.2. The following checks are normally assessed by a secondary reviewer (supervisor or QA Officer), but may be evaluated by the primary analyst:

1. Standard Reference Material
2. QC Check Samples
3. Spiked samples (matrix and blank)
4. Duplicates

11.2.3. The following must be assessed by the organization or individual(s) responsible for sample collection, but may be reviewed by laboratory personnel if the sample has been identified as:

1. Precleaned and field cleaned equipment blanks
2. Trip blanks
3. Field collected duplicates
4. Split samples

11.3 DETERMINING THE SOURCE OF QC PROBLEM

Once a problem has been identified, the process (whether analytical or review) should be halted until the reason for the problem has been identified. Finding the source of a QC problem involves identifying probable sources of error, and checking each source to determine if the protocols were properly followed. Common sources of error and expected follow-up protocols are outlined on Table 11-2. Usually, the individual who is responsible for identifying the problem is responsible for determining the cause. However, other personnel and organizations may need to cooperate.

11.4 INITIATING CORRECTIVE ACTION

When the source of a QC error has been identified, appropriate steps must be taken to eliminate or minimize recurrences.

11.4.1. If a QC measure listed in 11.2.1 above is not acceptable, testing cannot continue until the QC check meets specifications. Corrective actions may be initiated:

1. By the individual who is operating the instrument; or
2. By an individual in oversight authority (i.e. supervisor or QA Officer) if a solution is not immediately apparent.

11.4.2. Corrective actions for QC measures in 11.2.2 and 11.2.3 must be initiated by the individual who identifies the problem.

11.5 SPECIFIC CORRECTIVE ACTIONS

A list of expected corrective actions for each QC measure is included on Table 11-2. Since many QC problems have unique solutions, the corrective action protocols are not limited to those listed. Further assessment, based on an individual's experience and knowledge may be warranted.

11.6 DOCUMENTATION AND NOTIFICATION OF AFFECTED PARTIES

If a quality control measures fails to meet acceptance criteria, the QC measure, and the procedures were used to correct the problem must be documented.

Documentation does not imply a formal memo or corrective action form:

1. Corrective actions that are initiated during an on-going analytical run may be documented on the chromatogram, integrator or strip chart recorder records as well as in the instrument, analytical and/or field logs.

2. Corrective actions that require input or intervention of more than one individual must at a minimum be documented in the related logs and records. Corrective action forms for larger organizations are recommended.

3. If more than one organization is involved with identifying a QC problem and the associated corrective actions, formal memos are recommended, although dated and signed phone logs are acceptable. In all cases, a copy of all documentation should be maintained in the project files.

If an identified quality control problem affects more than one set of data or multiple projects, the documentation associated with identifying and resolving the problem must be cross referenced to all associated projects.

11.7 CORRECTIVE ACTIONS FROM EXTERNAL SOURCES

The need to initiate corrective action may be the result of activities or audits from external sources. Sources include systems audits; performance audits; split samples; blind QC samples; and findings from project or data validation review.

IN ALL CASES, DER RECOMMENDED CORRECTIVE ACTIONS MUST BE INITIATED.

TABLE 11.1
 ACCEPTANCE CRITERIA AND CORRECTIVE ACTIONS FOR
 QUALITY CONTROL CHECKS

QC CHECK	ACCEPTANCE CRITERIA
BLANKS	
Method blank	≤MDL
Reagent blank	
Calibration blank	
Precleaned Equipment	
Blanks	
Field Cleaned Equipment	
Blanks	
Trip Blanks	
CALIBRATION	
Initial calibration	a. Method acceptance criteria* b. DER SOP acceptance criteria
QC Check Standard	a. Method acceptance criteria* b. ± 10% of true value c. Internally generated control limits
Continuing calibration	a. Method acceptance criteria calibration* b. ± 20% deviation from initial calibration c. Internally generated control limits
Interference standard	Method acceptance criteria
Tuning criteria	Method acceptance criteria
SYSTEM PERFORMANCE CHECKS	
Pesticide	Method acceptance criteria
Standard Reference Materials	Within certified limits

* Method acceptance criteria shall be followed. If none is provided, the DER criteria (b) or internal, historically generated control limits (c) shall be used.

TABLE 11.1, continued
ACCEPTANCE CRITERIA AND CORRECTIVE ACTIONS FOR
QUALITY CONTROL CHECKS

QC CHECK	ACCEPTANCE CRITERIA
SYSTEM PERFORMANCE CHECKS, continued	
QC Check Samples	Within specified limits
SPIKES	
Matrix Spike	Within range specified by CompQAP (accuracy acceptance limits)
Blank Spike	Within method specified criteria
DUPLICATES	
Laboratory Duplicates	Within range specified by CompQAP (precision acceptance limits)
Matrix Spike Duplicates	Within range specified by CompQAP (precision acceptance limits)
Field Duplicates	Within range specified by CompQAP (precision acceptance limits)
OTHERS	
Surrogate Standards	Method acceptance criteria
Internal Standards	Method acceptance criteria
Split Samples	Meets precision criteria of CompQAP
Titration Solutions	a. $\pm 10\%$ of expected (lab determined) value b. Replicate sample aliquot results are within method specified limits
MICROBIOLOGY	
Monthly parameters Chlorine Residual Conductivity Heterotrophic Count	Per Page 305 of EPA-600/8-78-017
Annual metals concentration	Per Page 305 of EPA-600/8-78-017

TABLE 11.1, continued
ACCEPTANCE CRITERIA AND CORRECTIVE ACTIONS FOR
QUALITY CONTROL CHECKS

QC CHECK	ACCEPTANCE CRITERIA
MICROBIOLOGY, continued	
Distilled water suitability test	Per Table IV-A-3 of EPA-600/8-78-017
Autoclave	Adequate sterilization as determined by indicators
Incubators	35 \pm 0.5 C or 44.5 \pm 0.2 C
Duplicates	Within calculated precision criteria
Morphological and Biochemical Confirmation	Per Table IV-A-5 of EPA-600/8-78-017
Positive and negative media controls	Per Table IV-A-4 of EPA-600/8-78-017
MF Blanks	<1
MPN dilution blanks	<1
Inhibitory residue	Must meet method criteria
Membrane Filter Analysis	Verified colonies must be coliforms
MPN Analysis	Verified colonies must be coliforms
BIOASSAY	
One set of dilution water controls	Method acceptance criteria
Reference toxicant	a. Per specified method criteria b. Outside historically derived control limits
SPECIES IDENTIFICATION	
Confirmation	98-99% of unidentified species are verified by independent expert

TABLE 11.2
PROBABLE SOURCES AND EXPECTED CORRECTIVE ACTIONS

1. BLANKS

- a. Sources and expected review procedures:
 - 1. Contaminated reagents - verify reagent sources
 - 2. Environmental Contamination (all sample collection, sample and analysis conditions) - review sampling handling protocols
 - 3. Improper or incomplete laboratory and/or field decontamination/cleaning procedures - review cleaning protocols
 - 4. Contaminated sample containers - verify source and storage conditions
 - 5. Contaminated source water - verify water sources
- b. Expected Corrective Actions:
 - 1. Review data with respect to reported contamination levels. If sample concentrations are near the reported blanks levels, reprocess (reextract or digest) associated samples or resample. If sample concentrations or the reporting levels are significantly higher than blanks, or contaminants are not detected in the samples, report the sample data and concentrations in blank.
 - 2. Take measures to eliminate future problems: discard reagents, revise protocols, perform preventative maintenance on system, adjust use of interfering chemicals (solvents, fuels, etc.).

2. CALIBRATION

- a. Sources and expected review procedures:
 - 1. Improperly prepared or outdated standards - review preparation logs for calculation/dilution errors and use of expired sources.
 - 2. Improperly prepared or outdated check standard - verify check standard
 - 3. Poor instrument response - determine if preventative maintenance is required
 - 4. Incorrect calculations - review and verify all calculations
 - 5. Contamination problems (see blanks above)
- b. Expected Corrective actions:
 - 1. Recalculate calibration curve
 - 2. Prepare fresh standards
 - 3. Recalibrate instrument
 - 4. Perform preventative maintenance
 - 5. Perform mass calibration and retune
 - 6. Reanalyze all samples bracketing those from previous ACCEPTABLE QC check through next acceptable QC check

TABLE 11.2, continued
PROBABLE SOURCES AND EXPECTED CORRECTIVE ACTIONS

2. CALIBRATION, continued
 7. Take measures to eliminate sources of contamination
3. SYSTEM PERFORMANCE CHECKS
 - a. Sources and expected review procedures:
 1. Pesticides:
 - Poor column performance - replace/repack column
 2. Standard Reference Materials and QC Check Samples:
 - a. Improper sample preparation or analysis - review all protocols associated with sample preparation and analysis
 - b. Incorrect dilutions or calculations - recheck all calculations
 - c. Contamination (see blanks above)
 - b. Expected Corrective Actions:
 1. Reanalyze all samples bracketing those from previous ACCEPTABLE QC check through next acceptable QC check
 2. Reprocess all samples associated with QC check sample or standard reference material (unless the problem is unique to processing of the check sample)
 3. Take measures to eliminate sources of contamination
4. SPIKES
 - a. Sources and expected review procedures:
 1. Error in calculation - review/recheck all calculations
 2. Error in preparing or using spike solutions - review all preparation and/or analytical logs (including sample preparation) for proper dilutions, solvents, buffers, etc.
 3. Outdated standards - review expiration dates and standard preparation logs
 4. Contamination problems (see blanks above)
 5. Poor instrument response - determine if preventative maintenance is required
 - b. Expected Corrective Actions:
 1. Take measures to eliminate contamination problems, reprocess if necessary
 2. Perform required maintenance and revise pm schedules
 3. Review preparation, calculation and record keeping to determine if additional training or more stringent protocols are needed
 4. If the laboratory has no historical data to show that the sample matrix produces consistently unacceptable (out of control) recoveries, and none of the sources discussed above are responsible for the problem, the sample must be reprocessed and reanalyzed. If reanalysis produces the same result, associated samples should be reported with qualified results. If results are different, all associated samples must be reprocessed for analysis.

TABLE 11.2, continued
PROBABLE SOURCES AND EXPECTED CORRECTIVE ACTIONS

5. **DUPLICATES**
 - a. Sources and expected review procedures:
 1. Non representative sample - review sample collection and/or sample processing protocols
 2. Error in calculations - recheck calculations
 3. Contamination problems (see blanks above)
 4. See matrix spikes above
 - b. Expected Corrective Actions:
 1. Report data with qualifiers and explanation
 2. Revise sample collection/sample processing protocols to assure a representative sample
 3. Takes measures to eliminate contamination problems.
 4. Reprocess and reanalyze sample set (if laboratory generated replicate).
6. **SURROGATE SPIKES**
 - a. Sources and expected review procedures:
 1. See 4.a above
 - b. Expected Corrective Actions:
 1. See 4.b above
7. **INTERNAL STANDARDS**
 - a. Sources and expected review procedures:
 1. See 4.a above
 - b. Expected Corrective Actions:
 1. See 4.b above
 2. Reanalyze samples from last acceptable QC check to next acceptable QC check
9. **SPLIT SAMPLES**
 - a. Sources and expected review procedures:
 1. See 5.a above
 - b. Expected Corrective Actions:
 1. See 5.b above
10. **TITRATING SOLUTIONS**
 - a. Sources and expected review procedures:
 1. Error in calculation - review/recheck all calculations
 2. Error in preparing or using titrant and standard solutions - review all preparation and/or analytical logs (including sample preparation) for proper dilutions, solvents, buffers, etc.
 3. Outdated standards and/or - review expiration dates and standard preparation logs
 4. Contamination problems (see blanks above)
 5. Non representative sample - review sample collection and/or sample processing protocols

TABLE 11.2, continued
PROBABLE SOURCES AND EXPECTED CORRECTIVE ACTIONS

10. TITRATING SOLUTIONS, continued
 6. Indistinct or inconsistent endpoint readings
 - b. Expected Corrective Actions:
 1. Take measures to eliminate contamination problems, reprocess if necessary
 2. Review preparation, calculation and record keeping to determine if additional training or more stringent protocols are needed
 3. If replicate analyses are not acceptable, titrate additional aliquots
 4. Reanalyze samples from last acceptable QC check to next acceptable QC check
 5. Train analysts to titrate to consistent endpoint
11. MICROBIOLOGY - MONTHLY PARAMETERS, METALS, WATER SUITABILITY
 - a. Sources and expected review procedures:
 1. Deionizer/Water not functioning properly
 - b. Expected Corrective Actions:
 1. Clean, replace cartridges and/or perform other preventative maintenance tasks
 2. Reanalyze water
 3. Reprocess samples (if still within holding times) or resample
12. MICROBIOLOGY - AUTOCLAVE
 - a. Sources and expected review procedures:
 1. Autoclave not functioning properly
 - b. Expected Corrective Actions:
 1. Perform preventative maintenance and resterilize
13. MICROBIOLOGY - INCUBATORS
 - a. Sources and expected review procedures:
 1. Incubator not functioning properly
 2. Thermometers or recording devices not functioning properly
 - b. Expected Corrective Actions:
 1. Perform preventative maintenance on devices, recalibrate if necessary
 2. Reprocess samples (if still within holding times) or resample
14. MICROBIOLOGY - DUPLICATES
 - a. Sources and expected review procedures:
 1. Counting errors or difficulties in identifying coliform organisms (membrane filter).
 2. Nonrepresentative sample.
 3. Contamination problems.

TABLE 11.2, continued
PROBABLE SOURCES AND EXPECTED CORRECTIVE ACTIONS

14. MICROBIOLOGY - DUPLICATES, continued
 - b. Expected Corrective Actions:
 1. Recount or re-examination colonies to determine counting error or misidentifications.
 2. Examine blanks and samples analyzed to determine possible sources of contamination.
 3. If 14.a.2 above is found to be the problem, or the problem has not been identified, data must be invalidated and resampling and retesting must occur.
15. MICROBIOLOGY - ATCC AND BIOCHEMICAL CONFIRMATION
 - a. Sources and expected review procedures:
 1. Media prepared improperly (incorrect pH, sterilized too long, etc.).
 2. Incorrect incubator temperatures.
 3. Media shelf life has expired and no longer functions properly.
 - b. Expected Corrective Actions:
 1. Isolate problem with media preparation.
 2. Confirm proper incubator temperatures.
 3. Prepare new batch of media from the same lot to determine media acceptability or discard media if the shelf life has expired.
 4. Prepare new media from a different lot number.
 5. Invalidate all affected data linked to the media that was not functioning acceptability.
16. MICROBIOLOGY - POSITIVE AND NEGATIVE MEDIA CONTROLS
 - a. Sources and expected review procedures:
 1. See 15.a.1-3 under ATCC or biochemical confirmation.
 2. Samples used were not positive or negative.
 3. Improper analytical protocol.
 4. Contamination problems (negative control).
 5. Colony misidentification.
 6. Stressed organisms that did not respond in a typical fashion.
 - b. Expected Corrective Actions:
 1. See 15.b.1-4 under corrective actions for ATCC or biochemical confirmation.
 2. Re-examine response for misidentifications.
 3. Use alternate positive and negative control samples to confirm media response and check on original samples.
 4. Invalidate data and retest if problem was with the media or testing system.

TABLE 11.2, continued
PROBABLE SOURCES AND EXPECTED CORRECTIVE ACTIONS

17. MICROBIOLOGY - MF BLANKS

- a. Sources and expected review procedures:
 - 1. Equipment or rinse water/dilution water improperly sterilized.
 - 2. Rinsing technique not adequate.
 - 3. Contamination problems.
- b. Expected Corrective Actions:
 - 1. Review sterility checks on the autoclave for rinse/dilution water and other associated equipment.
 - 2. Evaluate rinsing protocols between samples.
 - 3. Review testing procedures and test location for other sources of contamination.
 - 4. Reject data and resample

18. MICROBIOLOGY - MPN DILUTION BLANKS

- a. Sources and expected review procedures:
 - 1. Equipment or dilution water not properly sterilized.
 - 2. Contamination problems during test procedure.
- b. Expected Corrective Actions:
 - 1. Review sterility checks on the autoclave for dilution water and other sterilized equipment.
 - 2. Review testing procedures and location for possible sources of contamination.

19. MICROBIOLOGY - INHIBITORY RESIDUE TESTS

- a. Sources and expected review procedures:
 - 1. Detergent residues inhibit bacterial growth.
 - 2. Alternate rinsing practice alleviates the problem.
- b. Expected Corrective Actions:
 - 1. Implement rinsing protocols that produce an acceptable inhibitory residue test.
 - 2. Change detergents to one that produces an acceptable test result under normal rinsing operations.

20. MICROBIOLOGY - MEMBRANE FILTER VERIFICATION

- a. Sources and expected review procedures:
 - 1. Compare original counts against verified colony counts.
- b. Expected Corrective Actions:
 - 1. Adjust initial colony count based upon positive verification percentage and report as verified coliform count.

21. MICROBIOLOGY - MPN COMPLETED TESTS

- a. Sources and expected review procedures:
 - 1. Compare original results against completed test results.

TABLE 11.2, continued
PROBABLE SOURCES AND EXPECTED CORRECTIVE ACTIONS

21. MICROBIOLOGY - MPN COMPLETED TESTS, continued
 - b. Expected Corrective Actions:
 1. Adjust original MPN result calculated from the completed test results.
22. BIOASSAY - DILUTION WATER CONTROLS
 - a. Sources and expected review procedures:
 1. See 1.a.1-5 above
 - b. Expected Corrective Actions:
 1. Invalidate data and retest
23. BIOASSAY - REFERENCE TOXICANTS
 - a. Sources and expected review procedures:
 1. Inappropriate test organisms
 - b. Expected Corrective Actions:
 1. Use new test organism or report data with qualifiers
24. SPECIES IDENTIFICATION
 - a. Sources and expected review procedures:
 1. Outdated identification keys
 2. Insufficient training in identification techniques
 - b. Expected Corrective Actions:
 1. Reidentify entire set and rerun associated data analyses
 2. Provide additional training
 3. Obtain updated keys

12.0 PERFORMANCE AND SYSTEMS AUDITS

12.1 FIELD AUDIT REQUIREMENTS

[[12.1.1 Internal Audits

1. Internal systems audits are conducted by company personnel in response to unacceptable or questionable QC or sample data and is a review and evaluation of the various components of the measurement and sample collection procedures to determine their proper selection and use. It should consist of a detailed review of each component (such as decontamination, meter and sampler calibration, field measurements, matrix sampling, Quality Control measures, documentation, sample custody, etc.)

- a. Systems audits should be conducted at least annually.
- b. Usually, these audits are conducted by the Quality Assurance Officer or his or her designee.
- c. A determination should be made that each element of an activity is functioning appropriately and within the guidelines of the proper methodology, the approved procedures and QA Plan.
- d. A list of deficiencies that must be addressed to correct, improve and modify the system must be generated as an end result.

2. Internal performance audits on field activities are not required by DER. If utilized, they should be documented as to the supplier of the sample, the type of sample used, and results should be included in all internal or external QA reports.]]

12.1.2 External Audits

1. External systems audits may be conducted by outside organizations. THE ADOPTION OF THIS SOP IS CONSIDERED TO BE A STATEMENT OF SUBMISSION TO ON-SITE DER AUDITS.

2. External performance audits should document the type of performance sample used in the audit and results should be included in all internal or external QA reports.

12.1.3 Project Audits - Data Validation by DER

Project-specific audits are conducted at the request of the DER project manager or when the project has been conducted without meeting the proper QA requirements. These audits consist of a complete review of the pertinent data, ancillary records and method validation packages.

12.2 REQUIREMENTS FOR AUDITS OF LABORATORY OPERATIONS

12.2.1 External Audits

12.2.1.1 Obligations for External Audits by DER

All parties are subject to facility, and data audits conducted by authorized representatives of DER. THE LABORATORY SHALL SUBMIT TO ANY SUCH AUDITS THAT HAVE BEEN REQUESTED BY DER OFFICIALS.

The DER audits may be a systems project or performance audit and will be conducted to determine compliance with the associated CompQAPs or QAPPs, Chapter 17-160, F.A.C., or other Department requirements (i.e., permits, consent orders, etc.).

12.2.1.2 Documentation and Reporting Requirements

In addition to DER audits, the laboratory may be subject to audits from other outside sources (e.g., EPA, HRS, etc.). All external audits shall be documented:

- a. Record the date and organization conducting all external system audits.
- b. Record the date and type of all external performance evaluations conducted by outside auditing parties.
- c. Summaries of all external audits shall be included in QA Reports to DER (see Section 13.0).

12.2.2 Internal Audits

[[12.2.2.1 Internal Systems Audit

Internal systems audits should be conducted as the complement to implementation and use of internal SOPs and Quality Plans, in order to assure good Quality Assurance management practices.

In general, procedures for conducting internal audits should be developed according to the following guidelines:

- a. Schedule systems audits to occur with routine frequency. Annual auditing of all lab operations is a minimum recommendation. Audits of selected systems may be staggered throughout the year to accomplish this goal.
- b. Develop a standardized protocol and list of minimum requirements which will constitute the style and scope of the audit and which will provide the criteria list by which operational deficiencies can be detected. These protocols and criteria should reflect the intent of all internal SOPs

and Quality Plans, and should at a minimum conform to all DER regulatory requirements for procedures and documentation. The use of standardized audit forms and checklists is recommended.

c. Designate appropriate personnel as Quality Assurance staff and charge these officials with auditing responsibility and authority, preferably independently of and lateral to the chain of authority responsible for laboratory operations.

d. Encourage all staff members to adopt good Quality Assurance practices, at all levels of the organization and to perceive audits as an educational opportunity.

The scope of internal systems audits of lab operations should include, but is not limited to the proper execution of:

- a. Electronic and paper documentation and filing associated with sample and data handling and all ancillary or support procedures, to include procedures employed to track all records pertinent to any sample results.
- b. All sample log-in, trafficking, log-out and disposal.
- c. Sample preparations.
- d. Calibrations.
- e. Sample analyses.
- f. Data reduction, validation and reporting.
- g. Standard and reagent preparation and storage.
- h. Waste disposal and segregation.
- i. Non-contaminating practices and the design/maintenance of non-contaminating laboratory environments.
- j. Container and labware decontamination and storage.
- k. Preventative maintenance and repair procedures.
- l. QC management practices and assessment of analytical precision, accuracy and sensitivity.
- m. proper promulgation and execution of established written procedures.]]

12.2.2.2 Requirements For Internal Performance Audits

Conduct blind, internal performance audits on all analytical systems employed to report data used by DER. These audits shall be conducted at least semiannually.

General requirements for internal performance audits are:

- a. Obtain QC samples of certified assay from external sources or vendors, or prepare QC check samples internally.

b. Samples may be composed in artificial matrices such as analyte-free laboratory water or in other matrices whose characteristics are well delineated and can be consistently controlled from sample to sample.

c. Analysts whose systems are to be audited shall not be made aware of the concentration values or the compound names of constituents of the audit sample. They may be informed of the nature of the samples or the audit samples may be inserted into the routine laboratory sample analysis train without the knowledge of the affected analysts.

d. Replicated analysis of the audit samples is discretionary. However, all routine QC procedures and sample handling procedures must be followed when analyzing performance samples. This is required in order that the performance audit may best represent the actual routine operations for the system.

12.2.2.3 Documenting and Reporting Internal Audits

Document all aspects of the audit. Retain all standard forms used in the audit, as well as all notes and final reports. Distribute audit reports or deficiency lists and corrective action orders to appropriate management staff affected, and verify execution of satisfactory corrective actions with follow-up documentation (see Section 11). Provide copies of all of the above to all staff at all levels involved in the audit or whose system area was affected. The following may also be included in all documentation and reports:

- a. Audit dates.
- b. Auditor names.
- c. Systems audited.
- d. Parameters analyzed in performance audits.
- e. Analysts involved in performance audits.
- f. Personnel interviewed for systems audits.
- g. All supporting documentation solicited or submitted in support of any systems, performance or data-package audit.
- h. Narrative description or report of findings, including summary charts and tables.
- i. Report condensations for executive summaries.
- j. Statistical evaluation report for performance audit analytical results.
- k. Recommended or required corrective actions.
- l. List of personnel for report distribution and follow-up responsibilities associated with corrective actions.

12.3 REGULATORY REQUIREMENTS

1. All field and laboratory audits are subject to the requirements outlined in Rules 17-160.650 and 17-160.680, F.A.C., of the QA Rules.

2. All project audits are subject to the requirements outlined in Rules 17-160.660 and 17-160.680, F.A.C., of the QA Rules.

3. All audits shall be reported in Quality Assurance Reports to DER (see Section 13).

13.0 QUALITY ASSURANCE REPORTS

Quality assurance reports are designed to keep project members informed of the performance of QA/QC activities. The reports should include all subjects which address the validity and documentation of data gathering activities. They summarize project specific audits, list significant problems, and discuss the solutions and corrective actions implemented concerning QA/QC activities.

13.1 QA REPORTS TO DER:

Quality assurance reports shall be submitted for all DER-QA Category 4 projects (Quality Assurance Project Plans). These reports shall be prepared and submitted to the Florida Department of Environmental Regulation Quality Assurance Section according to the following guidelines, unless specifically modified by contract, permit, or consent order decree.

Projects for which no audits were performed and no significant QA/QC problems occurred shall have a letter submitted asserting the following:

1. No Project Audits were performed (if applicable);
2. All sampling and support equipment were used as listed in the approved QAPP;
3. All preservation and holding time requirements have been met;
4. All field QC Blanks and duplicate results are within acceptable ranges; and
5. All analytical requirements for precision, accuracy, and MDL/PQL have been met.

13.2 REPORT FORMAT AND CONTENT:

13.2.1 Requirements for a Full Report

A project shall have a full report submitted if any of the following occurred:

1. Sampling and support equipment other than that specified in the approved QAPP were used;
2. Preservation or holding time requirements for any sample were not met;
3. Any quality control checks (field and laboratory) were unacceptable;
4. Any analytical requirements for precision, accuracy, or MDL/PQL were not met;
5. Sample collection protocols or analytical methods specified in the QAPP were not met;

6. Corrective action on any problem were initiated;
7. An internal or external systems or performance audit was conducted; or
8. Any other activity or event affected the quality of the data.

13.2.2 Contents

The QA reports shall contain the information listed below, and shall follow the outlined format:

1. Title Page - The following information must be listed:
 - a. Time period of the report
 - b. QA Project Plan Title and Plan number
 - c. Consultant/Laboratory Name, address and phone number
 - d. Preparer's name and signature
(See Fig. 13.1 for an example)
2. Table of Contents - Should be included if the report is more than ten pages long.
3. Audits - In table form, summarize all project specific audits that were performed during the specified time period (see Fig. 13.2 for an example):
 - a. Performance audits must include the following:
 1. Date of the audit
 2. System tested
 3. Who administered the audit
 4. Parameters analyzed
 5. Reported results
 6. True values of the samples (if applicable)
 7. If any deficiencies or failures occurred, summarize the problem area and the corrective action.
 - b. Systems audits must include the following:
 1. Date of the audit
 2. System tested
 3. Who administered the audit (agency or company)
 4. Parameters analyzed
 5. Results of tests
 6. Parameters for which results were unacceptable (include the reported and true values, if applicable)
 7. Explanation of the unacceptable results. Include probable reasons and the corrective action.
 - c. Copies of documentation such as memos, reports, etc. shall be enclosed.
4. Significant QA/QC Problems
 - a. Identify the problem, and the date it was found.
 - b. Identify the individual who reported the problem.
 - c. Identify the source of the problem.
 - d. Discuss the solution and corrective actions taken to eliminate the problem.

Fig. 13.1

TITLE PAGE FOR QA REPORT TO DER

QUALITY ASSURANCE REPORT TO DER

For the Time Period:

[Date] to [Date]

[Quality Assurance Project Plan Title and Number]

Prepared by:

[Consultant/Laboratory Name]
[Street Address]
[City, State and Zip Code]
[Phone Number]

Preparer's Signature
[Preparer's Name and Title]

Date

XYZ Laboratories, Inc.
Project Audit Report
January 1992 - January 1993

Performance Audit:

<u>Audit Date</u>	<u>Type</u>	<u>System Tested</u>	<u>Reported Results</u>	<u>True Value</u>	<u>Corrective Action</u>
2-92	Internal	Trace Metals for: Arsenic	12.5 ug/l	11.0 ug/l	Problem trace to improperly labeled calibration standards. Analyst reminded that all standards must be properly labeled with dilution factor and concentration. Proper documentation being checked daily.
4-92	Internal	VOCs for: Methylene Chloride	15.0 ug/l	1.0 ug/l	Problem traced to open bottle of methylene chloride left outside hood in extraction room. Technicians reminded that all solvents must be used under hood. Appropriate procedures are being monitored daily.
8-92	Internal	Blind Split Sampled with OHS Laboratory: XYZ Benzene Toluene Ethyl benzene	ONS 3.3 ug/l 5.0 ug/l 2.5 ug/l	ONS 3.7 ug/l 6.0 ug/l 2.1 ug/l	
1-92	EPA "MP Series", all parameters analyzed:	Mercury	1.5 ug/l	13 ug/l	Problem due to undocumented dilution. Record sheets redesigned to account for any dilutions.
		Ethyl benzene	1.0 ug/l	22 ug/l	Internal standard response in the sample was out of acceptable range. Sample should have been reanalyzed. Analyst has been advised to check I.S. responses.

Systems Audit:

<u>Audit Date</u>	<u>System Tested</u>	<u>Auditors</u>	<u>Significant Findings</u>	<u>Corrective Action</u>
1-92	Field Collection	Internal	Crew member assigned to make field measurements had not been trained on the equipment. Inadequate supply of organic blank water.	All new field personnel will be trained on measurement techniques before they will be allowed to use the equipment in the field. Additional glass carboys have been ordered for the crews.
9-92	Laboratory	EPA Region IV	Documentation for storage of Chain of Custody samples is inadequate. QC check standards are not being used in metal section. Temperatures in incubators are not monitored.	Sample custodian appointed and sample logs for storage area has been initiated. The practice of analyzing at least one QC check standard per analysis run has been initiated. Daily logs for incubator temperatures have been initiated.
11-92	Field	DER	pH adjusted samples were not checked in the field.	Samplers have been provided with narrow range pH paper and additional reagents to perform pH checks in the field. Protocols have been written, outlining specific procedures for checking pH in the field.

EXAMPLE OF AUDIT SUMMARY TABLE

Fig. 13.2

5. Corrective Actions Status

- a. Discuss the effectiveness of all corrective actions taken during the specified time frame as well any initiated during the previous report period.
- b. Discuss any additional measures that may be implemented as the result of any corrective action.

13.3 FREQUENCY:

Quality Assurance Reports/Letters shall be submitted per the frequency listed on Table 13.1.

13.4 INTERNAL QA REPORTS:

At a minimum, information will be circulated as necessary to keep project members informed of the performance of QA/QC activities.

This may be provided in the appropriate form of communication (verbal, formal memorandums or reports) to insure sound QA/QC management practices.

Copies of memorandums and reported should be filed with the project information as well as written logs of verbal communications.

THESE ROUTINE INTERNAL QA REPORTS SHALL NOT BE SUBMITTED TO DER.

TABLE 13-1
 SUBMITTAL FREQUENCY FOR QUALITY ASSURANCE REPORTS TO DER

PROJECT TYPE	PROJECT DURATION	FREQUENCY
Research Projects (per 17-160.230)	Not applicable	Quarterly
On-going monitoring projects	when sampling frequency is: Weekly, biweekly or monthly OR	Semiannual
	when sampling frequency is: Bimonthly, quarterly, or semiannual	Annual
Consent Order, Contamination Assessment Studies, Remedial Feasibility Studies	Up to two years OR	One final report
	Greater than two years	One report every two years, and one final report
Direct contract for sampling or analysis	Not applicable	A quarterly summary report which addresses all projects performed during the quarter
Other projects not defined above	Up to two years	One final report
	OR	
	Greater than two years	One report every two years, and one final report

(Taken from 17-160.700, F.A.C., Table 6)

APPENDIX A Definitions

The following definitions are used by the DER Quality Assurance Section and EPA. QA Plan writers are encouraged to use these terms as defined. If a consultant or laboratory chooses to define these terms in any other way, such definitions must be included in the associated QA Plan.

Analytical Set: The basic unit for analytical quality control. Also known as sample set or analytical batch. The analytical set is defined as samples which are analyzed (or sampled together) with the same method sequence, the same lots of reagents and with the same treatment common to all samples. The samples must have been analyzed (or collected) within the same specified time period or in continuous sequential time periods. Samples in each set should be of similar composition.

Audits: A systematic check to determine the quality of the operation of some function or activity.

Performance Audits: Quantitative data are independently obtained for comparison with routinely obtained data in a measurement system. Examples of these audits are EPA performance evaluation programs, commercial performance evaluation programs, split sampling program involving at least two laboratories, blind spike samples.

Systems Audits: These are qualitative in nature and consist of an on-site review and evaluation of a laboratory or field operations quality assurance system and physical facilities for sampling, calibration and measurements.

Project Audits: These consist of an independent review of all sampling and analytical activity records that are associated with a specific project or event to determine if the resulting data are valid and acceptable. Enough documentation must be available so that a reviewer is able to reconstruct the history of the samples from time of sample collection (or sample container acquisition) through final results and sample disposal.

Calibration: Process by which the correlation between instrument response and actual value of a measured parameter is determined.

Calibration Curve: A curve which plots the concentration of known analyte standards against the instrument response to the analyte. Also known as a Standard Curve.

Calibration Standard: Solutions or dilutions of a substance or material with a verifiable accuracy which are used to evaluate the sample property of an unknown sample. In analytical terms, these standards are used to establish a calibration curve or standard instrument response factors.

Continuing Calibration Standard: Standards that are analyzed during an analytical set to verify the accuracy of the calibration curve.

Internal Standard: A compound having similar chemical characteristics to the compounds of interest but which is not normally found in the environment or does not interfere with the compounds of interest. A known and specified concentration of the standard is added to each sample prior to analyses. The concentration in the sample is based on the response of the internal standard relative to that of the calibration standard and the compound in the standard.

Confidence Level: The statistical probability associated with an interval of precision (or accuracy) values in a QC chart. The values of confidence intervals are generally expressed as percent probability. It is a commonly accepted convention that the result being tested is significant if the calculated probability is greater than 90 percent, and is highly significant if the probability is greater than 99 percent.

Data Quality: The totality of features and characteristics of data that bears on its ability to satisfy a given purpose. The characteristics of major importance are accuracy, precision, completeness, representativeness, and comparability. These characteristics are defined as follows:

Accuracy: The degree of agreement of a measurement (or an average of measurements of the same thing), X , with an accepted reference or true value, T , usually expressed as the difference between the two values, $X-T$, or the difference as a percentage of the reference or true value, $100 (X-T)/T$, and sometimes expressed as a ratio, X/T . Accuracy is a measure of the bias in a system. Accuracy shall be calculated according to the formulae in Section 9.2.3.2 and 9.2.3.3 of this Manual.

Precision: A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions." Precision shall be calculated according to the formulae listed in 9.2.3.1 of this Manual.

Representativeness: Expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Comparability: Expresses the confidence with which one data set can be compared to another.

Data Quality Objectives: A set of specifications that the environmental data must meet in order to be acceptable for its

intended use in a program area. DQOs are commonly established for limits of detection and quality of data (precision, accuracy, representativeness and comparability).

Detection Limits: The smallest concentration/amount of an analyte of interest that can be measured with a stated probability of significance. Detection limits must be further defined as:

Method Detection Limit: The smallest concentration of an analyte of interest that can be measured and reported with 99 percent confidence that the concentration is greater than zero. The MDLs are determined from the analysis of a sample in a given matrix containing the analyte at a specified level. Determination of MDLs must be done by procedures specified in Section 9.3 of this Manual. Equivalent procedures to determine MDLs must be approved by DER.

Practical Quantitation Limit: The smallest concentration of an analyte of interest that can be reported with a specific degree of confidence. PQLs shall be determined in the same way as MDLs by using the procedures specified in Section 9.2.1 of this Manual. The standard deviation (sd) derived from the procedures will be used to calculate the PQL: $PQL = 12 \text{ sd}$.

Instrument Detection Limit: The smallest amount of an analyte of interest that generates an instrument response (signal) under prescribed conditions such that the magnitude of the signal is larger than the absolute uncertainty (error) associated with it.

Environmental Sample: Means any sample from a natural source or source that may reasonably be expected to contribute pollution to or receive pollution from ground waters or surface waters of the state. This includes, but is not limited to: receiving waters; waters used to define natural background conditions; soils; sediments; industrial, domestic or municipal discharge effluents; chemical storage or handling facilities; waste disposal facilities or areas; industrial or agricultural chemical handling or application areas; surface water run-off; and facilities for handling or applying of chemicals for weed or insect control [definition per Rule 10D-41.101(7), F.A.C.].

Parent Sample: Refers to a sample from which aliquots are taken for testing purposes.

Subsample: Refers to any derivative obtained from a sample. These include, but are not limited to: aliquots; filtrates; digestates; eluates; fractions; extracts; reaction products; supernatants; etc.

Organizational Terms:

Internal: Refers to operations, personnel, documents and protocols within the specified organization.

External: Refers to operations, personnel, documents and protocols from a party that is separate from or outside the specified organization.

Parameter Group: Is defined as a group of samples that have been preserved in the same manner, prepared by similar protocols and analyzed using instruments of similar technology (also known as analyte group). Examples of parameter groups are:

- Volatiles (EPA methods 601, 602, and 624)
- Pesticides (EPA methods 608, 614, 622)
- Trace Metals (All metals except mercury)
- Nutrients (Total Kjeldahl Nitrogen, Nitrate + Nitrite, Total Phosphorous)

Performance Evaluation Samples: A sample submitted for analysis whose composition and concentration are known to the submitter but unknown to the analyst. Also known as a Blind Sample.

Quality Assurance: A system of activities whose purpose is to provide the producer or user of environmental data the assurance that it meets defined standards of quality with a stated level of confidence.

Quality Assurance Plans (QAP): An orderly assembly of detailed and specific procedures which delineates how data of a known and accepted quality is produced.

Comprehensive Quality Assurance Plan (CompOAP): A QA plan that outlines all the capabilities of the specified organization, the routinely used quality control measures, the routine QA targets for precision and accuracy, and all documentation, calibration and maintenance activities that are necessary to produce data of a known and acceptable quality.

Quality Assurance Project Plans (QAPP): A QA plan that is written for a specific project outlining specific QA targets and data quality objectives as well as all protocols and QC measures needed to meet the project specific objectives. A QAPP must be submitted using DER Form 17-160.900(1).

Research Quality Assurance Plans (ROAP): A special type of Quality Assurance Project Plan that is generally written as a requirement of a direct contract with DER for research activities. The specific activities are defined in section 6.0 of the Manual for Preparing QA Plans. The content and format requirements are different from those of any other QAP.

Quality Control: The overall system of activities whose purpose is to document and control the quality of environmental data so that it meets the needs of the users.

Quality Control Measures:

1) **Blanks:** An artificial sample of an analytical matrix designed to monitor the introduction of artifacts into the system.

a) **Field Quality Control Blanks**

1) **Field Blanks:** Blanks of analyte free water that are prepared on-site by filling appropriate sample containers with the water, adding appropriate preservatives, sealing the containers, and completing the appropriate documentation. These blanks should be prepared during the middle to end of a sampling event by filling sample containers with water from the equipment decontamination water transport containers. They are to be treated, stored, transported, and analyzed in the same manner as the sample group for which it was intended. These blanks may be submitted for all water parameter groups.

2) **Equipment Blank:** Blanks of analyte-free water that are prepared on-site by pouring the equipment decontamination water through decontaminated field equipment. Appropriate sample containers, for each analyte group must be used, preservatives added, if required, and appropriate documentation must be completed. These blanks are to be stored, transported and analyzed with the intended parameter groups. At least one equipment blank is required for each water and solid matrix analytical group, and must be collected at the beginning of the sampling episode. If field decontamination is performed on-site, additional equipment blanks must be submitted for all water and solid matrix analytical groups.

3) **Trip Blank:** These blanks are required for only VOC samples. Blanks of volatile organic free water that are prepared by the organization that is providing the sample containers. These are transported to the site with the empty VOC sample containers, and shipped to the analyzing laboratory in the same containers as the VOC samples. They remain unopened for the entire trip. Proper labeling and documentation must be completed. A trip blank must be submitted for each cooler that transports VOC samples.

b) **Laboratory**

1) **Method Blank:** A blank of an appropriate analyte-free matrix that is processed (digested, extracted, etc.) and analyzed with a specified sample set.

2) **Reagent Blank:** An aliquot of analyte-free water or solvent that is analyzed with a sample set.

2) **Spiked Samples:** Samples fortified to a known and validated concentration of analyte. Percent recoveries are calculated for each compound in the spike.

a) **Field:** An environmental sample fortified to a known and validated concentration in the field. These may be submitted as blind spike (laboratory does not know they are spiked) or as identified field spikes. The use of this QC check is not recommended by DER.

b) Laboratory:

1) Reagent Spikes: Samples of an appropriate analyte-free matrix (deionized water, sand, soil, etc.) that are fortified to a known and validated concentration of analyte(s) before sample preparation.

2) Sample (Matrix) Spikes: Environmental sample selected from a set (not blanks) that are fortified to a known and validated concentration of analyte(s) before sample preparation. The concentration of each analyte in the spiking solution should be approximately 3-5 times the level expected in the sample.

3) Surrogate Spikes: Samples fortified with a compound having similar chemical characteristics to the compounds of interest, but which is not normally found in environmental samples. Known concentrations of these compounds are added to all samples in the set before sample preparation.

3) Replicate Sample: Samples that have been collected at the same time from the same source (field replicates) or aliquots of the same sample that are prepared and analyzed at the same time (laboratory replicates). Duplicate samples are one type of replicate sample. The analytical results from replicates are used to determine the precision of a system. If the concentration of analytes in the sample are below detectable limits, Duplicate Spike Samples may be used to determine precision. Blind Replicates (Duplicates) are replicates that have been collected (field replicate) or prepared (laboratory replicate) and are submitted and analyzed as separate samples (analyst does not know they are replicates).

4) Quality Control Checks: Standards or samples from an independent source that are analyzed at a specified frequency.

a) Quality Control Check Standards: Standard solutions from a source other than normal calibration standards that are certified and traceable. These standards are used to check the accuracy of a calibration curve.

b) Quality Control Check Samples (also known as Reference Materials): Samples obtained from an independent source for which the level(s) of analytes have been validated. These samples are prepared and analyzed with a sample set of similar matrix. If these samples have been obtained from the National Institute of Standards and Technology (formerly National Bureau of Standards), these are referred to as Standard Reference Materials.

5) Split Samples: Replicates of the same sample that are given to two independent laboratories for analysis.

6) Acceptance Criteria: The numerical limits, prescribed by the approved analytical method or internal data, by which an analytical system is verified. These numerical limits may be generated from internal, historical data using the formula

specified in Section 9.2.3.4. Acceptance criteria shall be generated and used for all Quality Control Measures described above. Also known as Control Limits.

Sample Custody - All records and documentation required to trace a sample from point of origin through disposal after analysis. These records must include, but are not limited to:

- 1) Field notebooks;
- 2) Field sample ID tags;
- 3) Laboratory transmittal forms (if applicable);
- 4) Laboratory sample receipt logs;
- 5) Sample extraction/preparation logs or worksheets;
- 6) Analytical (instrument) logs or worksheets;
- 7) Calibration and quality control data associated with a sample set;
- 8) Instrument maintenance logs;
- 9) Sample disposition logs; and
- 10) Final reports.

Legal Chain of Custody is a special type of sample custody in which all events (i.e. possession, transport, storage, and disposal) and time intervals that are associated with a specific sample must be documented in writing. In addition to the records described above, chain of custody records must include the following:

- 1) Sample transmittal forms or tags that have adequate spaces for the dated, original signatures of all individuals who handle the sample (or cleaned sample containers if obtained from a contracted laboratory) from time of collection (or container receipt) through laboratory delivery.
- 2) Laboratory sample storage logs that identify date, time, and individuals who remove samples from storage.
- 3) Secure, limited access storage areas.

Sample Matrix means that characteristic of an environmental or laboratory sample, associated with its physical and chemical properties, which defines how such a sample is handled when subjected to the intended analytical process. The following samples matrices (major matrix groups), as defined below, should be used in QA plans whenever specifying data quality objectives:

Analyte-Free Water: Water in which all analytes of interest and all positive or negative interferences are below method detection limits. The absence of such components shall be documented by analytical records.

Reagent Water: A sample of water which conforms to ASTM grades II, III or IV.

Drinking Water: Includes finished (treated) or raw source water designated as potable water. Such sources may be from surface or ground water.

Surface Water: Includes fresh or saline waters from streams, canals, rivers, lakes, ponds, bays and estuaries (natural or manmade).

Groundwater: Includes all waters found below ground in confined or unconfined aquifers.

Wastewater: Includes any influent or effluent associated with domestic or industrial waste treatment facilities.

Chemical Waste: Includes sludges and residuals from domestic or industrial wastewater processing, and liquid or solid chemicals that are no longer used for its intended purpose.

Soil/Sediment: Surface or subsurface soils and sediments of fresh or salt water origin.

Biological Tissue: Includes tissues of plant or animal origin. The most common of these are shellfish, finfish and aquatic plants.

Sampling Kit: A set of sampling accessories that has been assembled for a specified use or project. A Sampling Kit may include, but is not limited to: sample containers; sampling equipment (e.g., bailers); sample preservatives, trip blanks; reagent transfer tool (e.g., disposable pipets); calibration standards; indicator papers (e.g., pH paper); or reagents. Sampling Kits shall be subject to the documentation outlined in Section 5.0.

APPENDIX B
Selected References

Quality Control/Quality Assurance

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4. "Limit of Detection - A closer Look at the IUPAC Definition", Analytical Chemistry 55, 712A - 718A (June 1983).
5. Manual for Analytical Quality Control for Pesticides and Related Compounds in Human and Environmental Samples. EPA 600/1-79-009. 1979.
6. "Nomenclature, symbols, units and their usage in spectrochemical analysis--II", Spectrochim. Acta B, 33B, 242 (1978)
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Technical

8. Annual Book of ASTM Standards. Vol. 11.01 and 11.02 (Water I and II). American Society for Testing and Materials. 1989.
9. Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual. U. S. EPA Region IV Environmental Services Division. February 1991.
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11. Manual for Certification of Laboratories Analyzing Drinking Water. EPA 570/9-90/008. April 1990.
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13. Methods for Chemical Analysis of Water and Wastes. EPA 600/4-79-020. Revised March 1983.
14. Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples. Book 5, Chapter A4. U. S. Geological Survey. 1977.
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21. Official Methods of Analysis of the Association of Official Analytical Chemists. 15th edition. AOAC. 1990.
22. Procedures for Handling and Chemical Analysis of Sediments and Water Samples. EPA/Corps of Engineers CE-81-1. March 1981.
23. RCRA Ground-water Monitoring Technical Enforcement Guidance Document, (TEGD). EPA OSWER-9950.1. September 1986.
24. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third edition. EPA/600/4-91/002. 1991.
25. Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Second edition. EPA/600/4-91/003. 1991
26. Standard Methods for the Examination of Water and Wastewater. 17th Edition. 1989.
27. Test Methods for Evaluating Solid Waste - Physical/Chemical Methods. EPA/SW 846. 3rd Edition, 1986 and Final Update I Dated November 1990.
28. USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis. EPA. SOW No. ILMO 2.1. September 1991.
29. USEPA Contract Laboratory Program Statement of Work for Organic Analysis. EPA. SOW No. ILMO 2.1. September 1991.

PROJECT PLAN REVISIONS

Navy RAC PP: 6026
Date: 01/16/96
Attachment: 6.1
Page: 1 of 1

Attachment 6.1 PROJECT PLAN REVISION

Document Name and No.	<u>Radiological Work Plan</u>		Rev.	<u>0</u>	PPR #	<u>608-001</u>
Site	<u>NAS Jacksonville</u>	Task	<u>1</u>	Phase	<u>3</u>	DO No.
Initiator	<u>Dale Obenauer</u>					
<p>Description <u>Contaminated soils will be removed to eliminate "hot spots" as designated in the PSC 9 Radiological Survey Report for Naval Air Station, Jacksonville, FL prepared by BEI in May 1996. Designated areas to be excavated as identified in the report are: 01-01A, 03-01A, 04-01A, 05-04, 06-01, 08-01, 10-02, and 13-01A. Maximum depth of excavation will be 2 feet below land surface as described in BEI's technical proposal 010710 dated 10 June 1997.</u></p> <p>Reason <u>Excavated soils from PSC 9 will be placed in trucks rather than containerizing soils in drums for offsite shipment and disposal. Excavated soils will be transported to PSC 26 for inclusion in the landfill prior to capping as directed by the Navy in the attached Modification to Statement of Work #35.</u></p>						

IMPACT REVIEW

FUNCTIONAL REP.	NO	YES	IDENTIFY AFFECTED DOCUMENTS
PE <u>7/11/97</u>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
S&H Supv. <u>7-22-97</u>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
QCM <u>7-17-97</u>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Site Super. <u>Steve Rhoda</u>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Other _____	<input type="checkbox"/>	<input type="checkbox"/>	

P. H. Davis 7/22/97
Project Manager Date

Steve Rhoda 8/5/97
Navy Technical Representative Date

Change Approved:

Trend Required:

Navy Concurrence Required:

Change Approved:

☐ No ☒ Yes

☒ No ☐ Yes

☐ No ☒ Yes

☐ No ☒ Yes

**MODIFICATION
TO
STATEMENT OF WORK #35**

**CONTRACT NO N62467-93-D-0936
DELIVERY ORDER NO. 0035**

The contractor shall perform the following modifications to the original statement of work:

4.2 Task 2 Removal of RAD Waste at PSC 9 and 15

4.2.1 Phase 3 The contractor shall perform those tasks necessary to execute the Radiological Remedial Work Plan (RRWP) for NAS Jacksonville, PSCs 9 and 15. The contractor will prepare a cost estimate for the removal, containerizing, transport of radioactive wastes at PSC 9 and 15. The contractor will perform removal, containerizing radioactive wastes at PSC 9 and 15. The contractor will transport and deposit those waste at PSC 26. Composite soil samples are required for TCLP analysis at this site. Radiological surveying of the site will be conducted to verify the extent of the contaminated soils and the verification of removal of soils as specified in the RRWP. Upon completion of the removal action, the contractor shall prepare a post-construction documentation report detailing what was accomplished.

Clear area for radiological removal. Clearing of area will be coordinated through NAS Jacksonville Environmental Engineering Office.

Establish Schedule and Updates - Provide work schedule and work description of radiological remedial activities and coordinate with the NAS Jacksonville Environmental Engineering Office (Diana Lancaster) to allow sufficient time to notify the general public and base personnel of the remedial action operations.

The proposal shall be structured so the cost of each PSC can be separated for accounting purposes.

PROJECT PLAN REVISIONS

Navy RAC PP: 6026
 Date: 01/16/96
 Attachment: 6.1
 Page: 1 of 1

Attachment 6.1
 PROJECT PLAN REVISION

Document Name and No.	Radiological Work Plan		Rev.	0	PPR #	609-001
Site	NAS Jacksonville	Task	1	Phase	3	DO No.
Initiator	Dale Obenauer					
<p>Description Contaminated soils will be removed to eliminate "hot spots" as designated in the <i>PSC 15 Radiological Survey Report for Naval Air Station, Jacksonville, FL</i> prepared by BEI in May 1996. Designated areas to be excavated as identified in the report are: 01-02A, 03-01A, and 3-02. Maximum depth of excavation will be 2 feet below land surface as described in BEI's technical proposal 010710 dated 10 June 1997.</p>						
<p>Reason Excavated soils from PSC 15 will be placed in trucks rather than containerizing soils in drums for offsite shipment and disposal. Excavated soils will be transported to PSC 26 for inclusion in the landfill prior to capping as directed by the Navy in the attached Modification to Statement of Work #35.</p>						

IMPACT REVIEW

FUNCTIONAL REP.	NO	YES	IDENTIFY AFFECTED DOCUMENTS
PE <i>OKO</i> 7/11/97	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
S&H Supv. <i>PSL</i> 7/22/97	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
QCM <i>MS</i> 7/24/97	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Site Super. <i>7-17-97</i> <i>Severhede</i>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	
Other _____	<input type="checkbox"/>	<input type="checkbox"/>	

Project Manager

Date

Change Approved:

☐ No ☒ Yes

Trend Required:

☒ No ☐ Yes

Navy Concurrence Required:

☐ No ☒ Yes

Change Approved:

☐ No ☒ Yes

Navy Technical Representative

Date

**MODIFICATION
TO
STATEMENT OF WORK #35**

**CONTRACT NO N52467-93-D-0936
DELIVERY ORDER NO. 0035**

The contractor shall perform the following modifications to the original statement of work:

4.2 Task 2 Removal of RAD Waste at PSC 9 and 15

4.2.1 Phase 3 The contractor shall perform those tasks necessary to execute the Radiological Remedial Work Plan (RRWP) for NAS Jacksonville, PSCs 9 and 15. The contractor will prepare a cost estimate for the removal, containerizing, transport of radioactive wastes at PSC 9 and 15. The contractor will perform removal, containerizing radioactive wastes at PSC 9 and 15. The contractor will transport and deposit those waste at PSC 26. Composite soil samples are required for TCLP analysis at this site. Radiological surveying of the site will be conducted to verify the extent of the contaminated soils and the verification of removal of soils as specified in the RRWP. Upon completion of the removal action, the contractor shall prepare a post-construction documentation report detailing what was accomplished.

Clear area for radiological removal. Clearing of area will be coordinated through NAS Jacksonville Environmental Engineering Office.

Establish Schedule and Updates - Provide work schedule and work description of radiological remedial activities and coordinate with the NAS Jacksonville Environmental Engineering Office (Diana Lancaster) to allow sufficient time to notify the general public and base personnel of the remedial action operations.

The proposal shall be structured so the cost of each PSC can be separated for accounting purposes.